

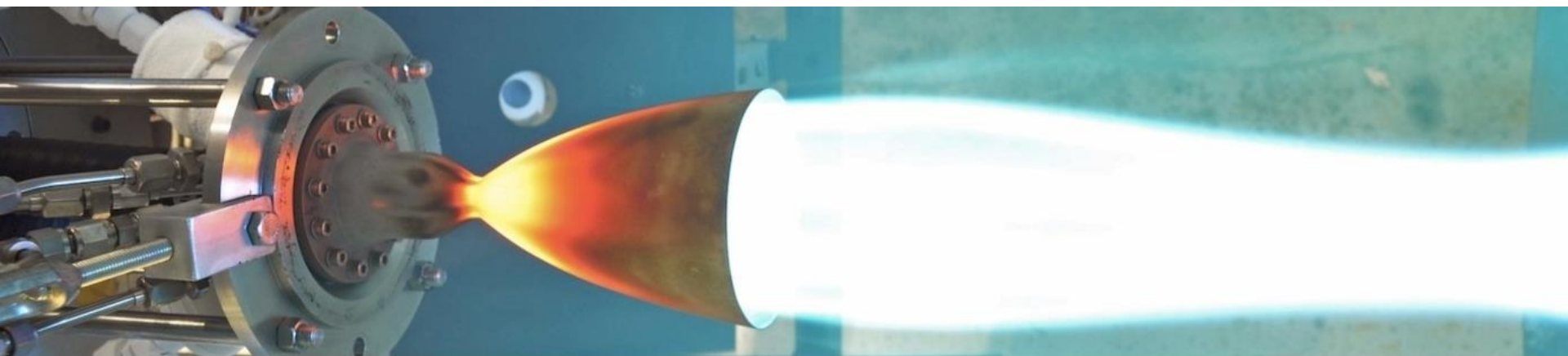


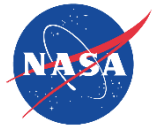
# GRC Webinar – Additive Manufacturing High Temperature Alloys

Dr. Tim Smith PhD

**LEW-20020-1:** “Novel Fabrication Technique for Oxide Dispersion Strengthened (ODS) Alloys”

**LEW-19886-1:** “Additively Manufactured Oxide Dispersion Strengthened Medium Entropy Alloys for High Temperature Applications”





## Tim Smith - Bio



2011: Graduated from Wright State University with a Bachelors degree in Mechanical Engineering

2016: Graduated from Ohio State University with a PhD in Materials Science

2015: Began career at the NASA Glenn Research center through the Pathways Internship Program

2016-Present: Currently works at GRC where his research focuses on the production, microstructural characterization, and deformation analysis of high temperature alloys.

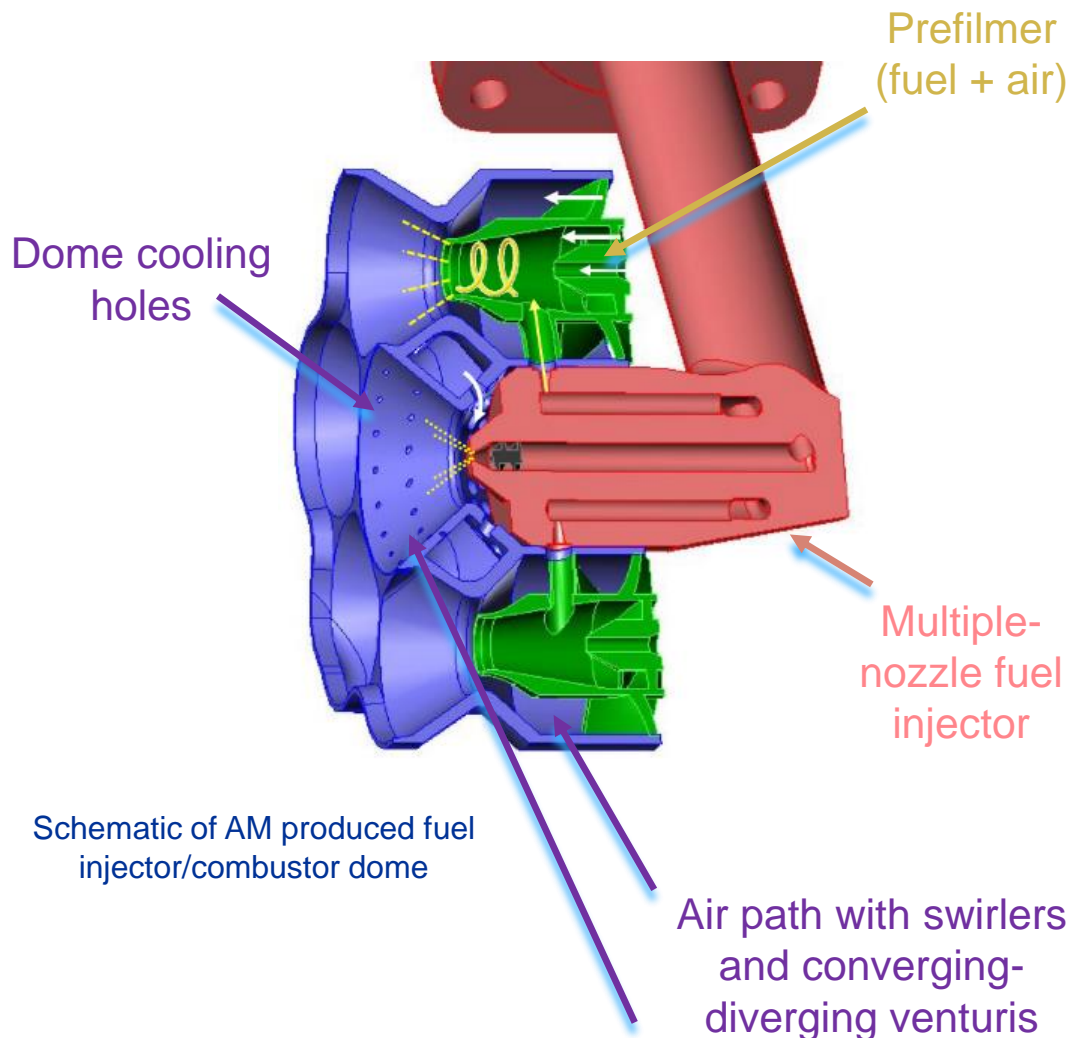
He has contributed to 29 peer-reviewed journal articles including publications in Nature Communications and Nature Communications Materials.

# Background – NASA Application

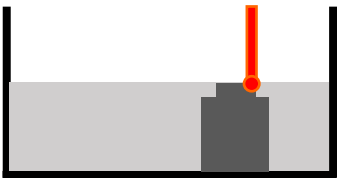
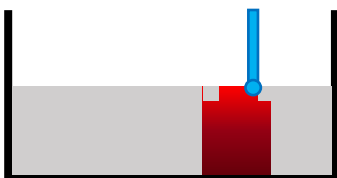
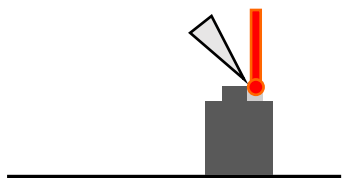
**Problem:** Conventional materials and processing techniques limit the design of combustor domes used in jet turbine engines.

**Proposed Solution:** Develop a high ductility, high temperature material for an additively-manufactured (AM) combustor fuel nozzle and dome for supersonic aircraft ( $>1093^{\circ}\text{C}$  ( $2000^{\circ}\text{F}$ ) operating temperature).

- Lead to several improvements to the turbine combustor design ultimately reducing NO<sub>x</sub> pollution and lowering weight.
- May enable lean-front-end small-core combustors.



# Metallic Additive Manufacturing

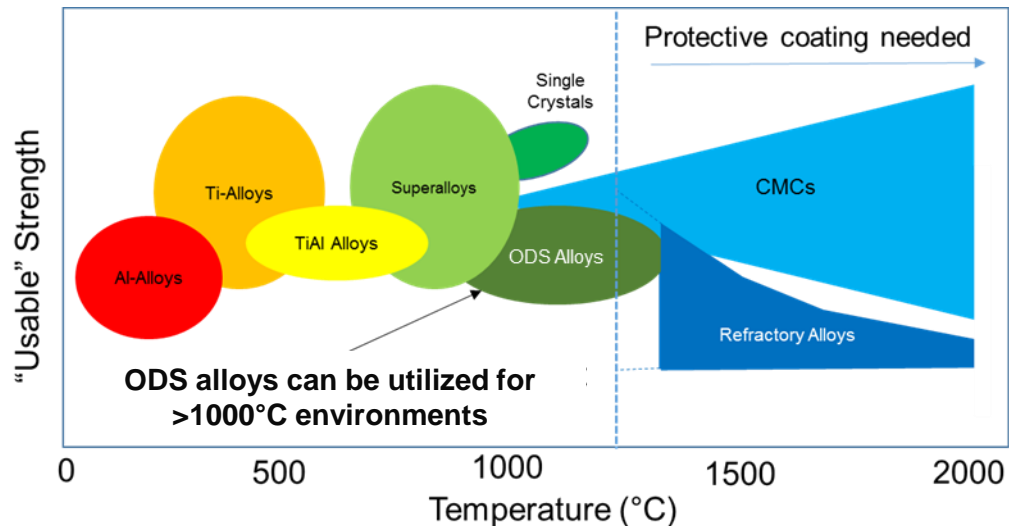
Process	Laser Powder Bed Fusion (L-PBF)	Electron Beam Powder Bed Fusion	Direct Energy Deposition (DED)
			
Energy Source	Laser	E-Beam	Laser or E-Beam
Powder Bed	Yes	Yes	No
Power (W or kV)	50-1000 W	<b>30-60kV</b>	100-2000 W
Max Build Size (mm)	500 x 280 x 320	500 x 280 x 320	<b>2000 x 1500 x 750</b>
Material	Metallic Powder	Metallic Powder	Metallic Powder or Wire
Dimensional Accuracy	<b>&lt;0.04 mm</b>	0.04-0.2 mm	0.5 mm (powder) 1.0 mm (wire)

- 3D printing or additive manufacturing (AM) has shown promise in realizing a new design space for aerospace applications.
- Each AM technique has a set of pros and cons associated with them.
- Instead of producing well known cast and wrought alloys with AM. We should look at AM as a new opportunity to produce materials that are currently difficult to create.
- For this study, L-PBF is used due to its superior dimensional accuracy.

# High Temperature AM Compatible Materials

## High Temperature Materials:

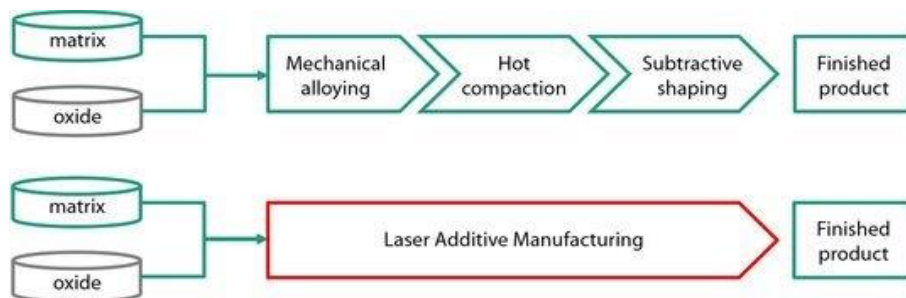
- Refractory metals
- Carbon-Carbon composites
- CMC's
- Ni-base superalloys
- **Oxide Dispersion strengthened (ODS) alloys**



Inspired by Andy Jones. ODS alloy Development.

(ODS) alloys offer higher temperature capabilities compared to Ni-base superalloys. However, it has been a challenge to produce ODS alloys through conventional manufacturing methods.

## Conventional Manufacturing vs AM



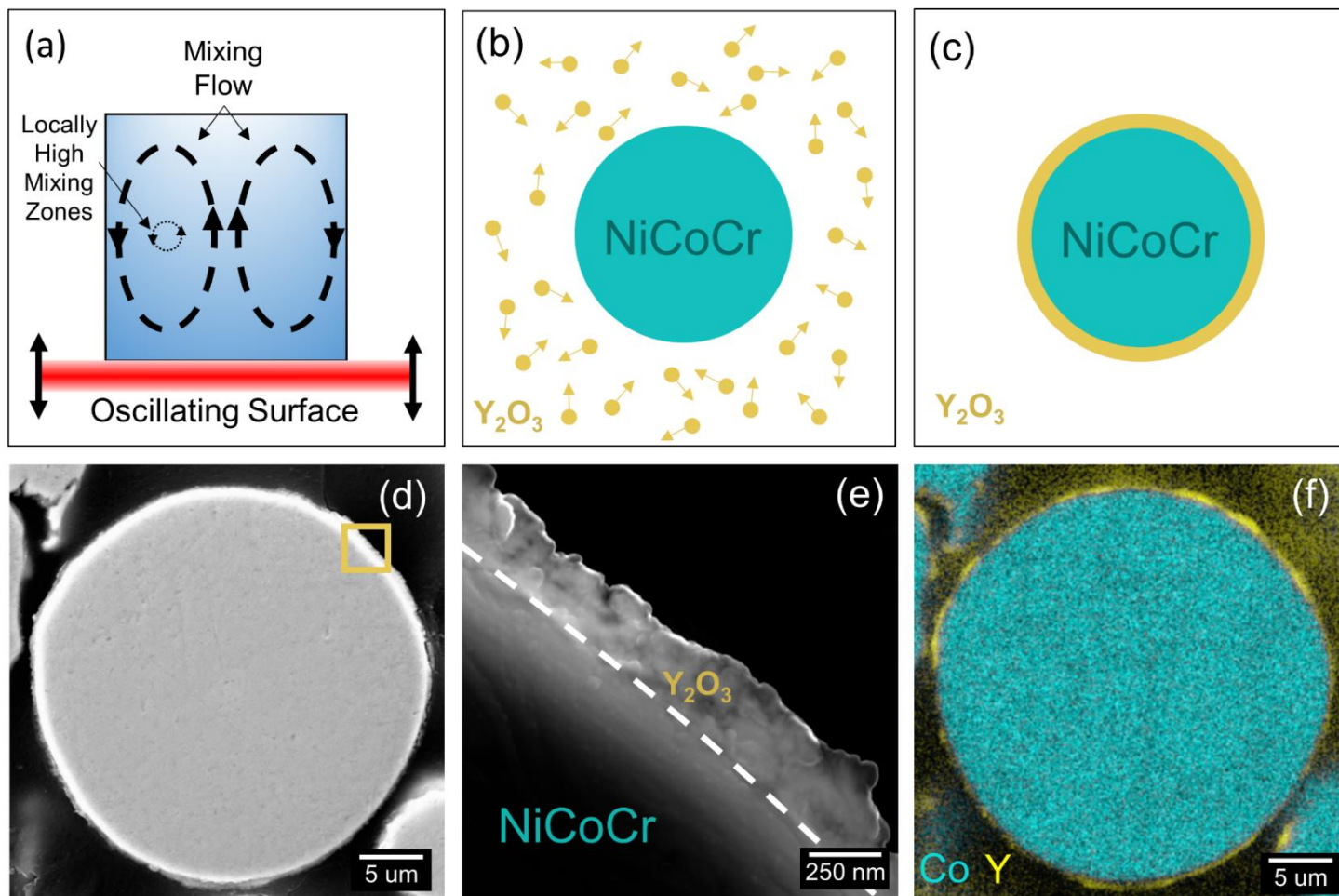
Can AM improve ODS alloy manufacturability?



# Methods

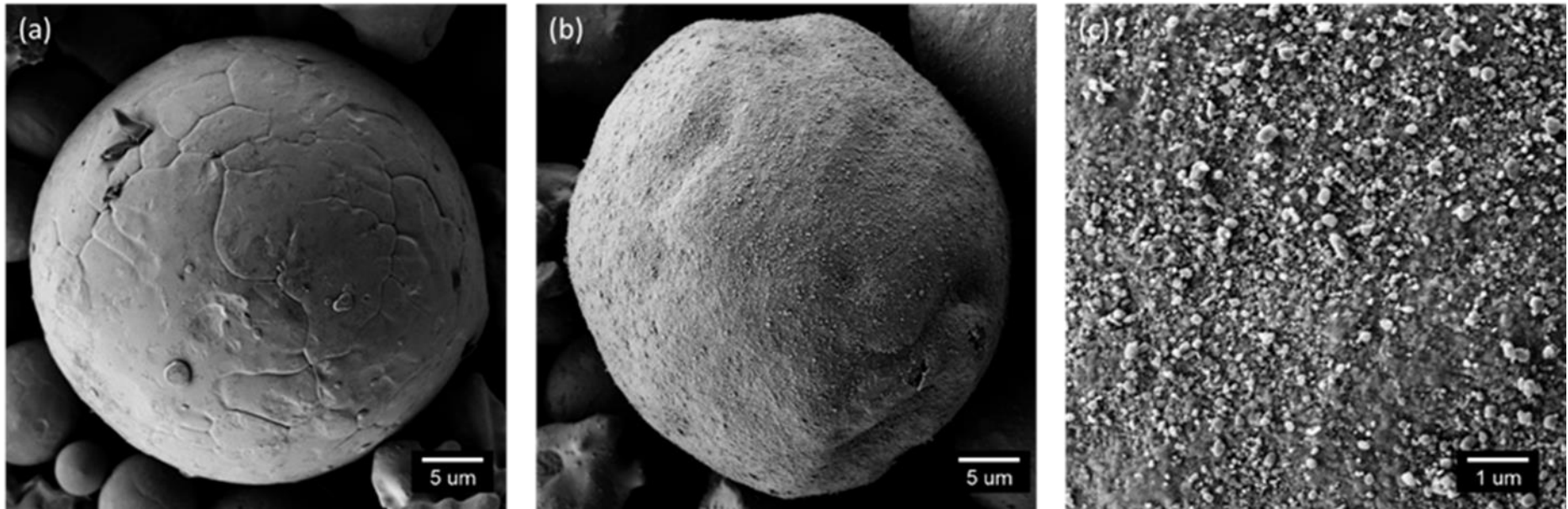
- Micron-scale (10-45um) NiCoCr and NiCoCr-ReB powder was acquired from Praxair.
- Nano-scale (100-200nm)  $Y_2O_3$  powder was acquired from American Elements.
- L-PBF Machine: EOS M100
- Powder Mixing: Resodyn LabRAM II
- Aim of study
  - Leverage L-PBF to produce oxide dispersion strengthened multi-principal element alloys.
  - Determine optimal L-PBF laser parameters for baseline AM NiCoCr, oxide dispersion strengthened ODS-NiCoCr, and ODS-NiCoCrReB (ODS-ReB) builds.
  - Produce 99.9% dense vertical test specimen for microstructural and mechanical analysis using AM NiCoCr, ODS-NiCoCr, and ODS-ReB.
  - Explore heat treatment effects on mechanical performance
  - Produce a high temperature capable 3D printed combustor dome.

# LEW-20020-1: “Novel Fabrication Technique for Oxide Dispersion Strengthened (ODS) Alloys”



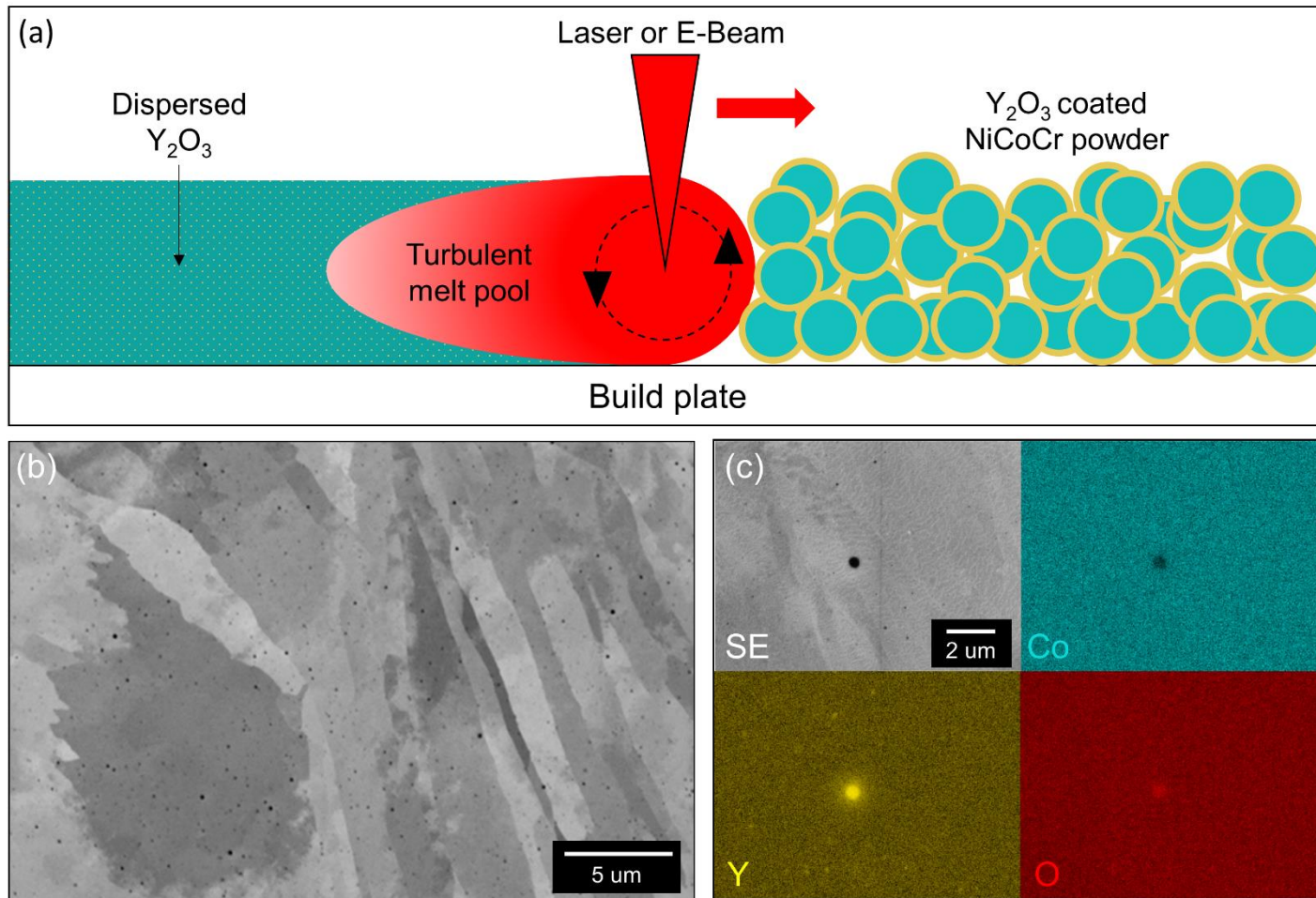
New high energy mixing technique successfully coats NiCoCr powder with 1 wt.%  $Y_2O_3$ .

# Novel Powder Coating Technique



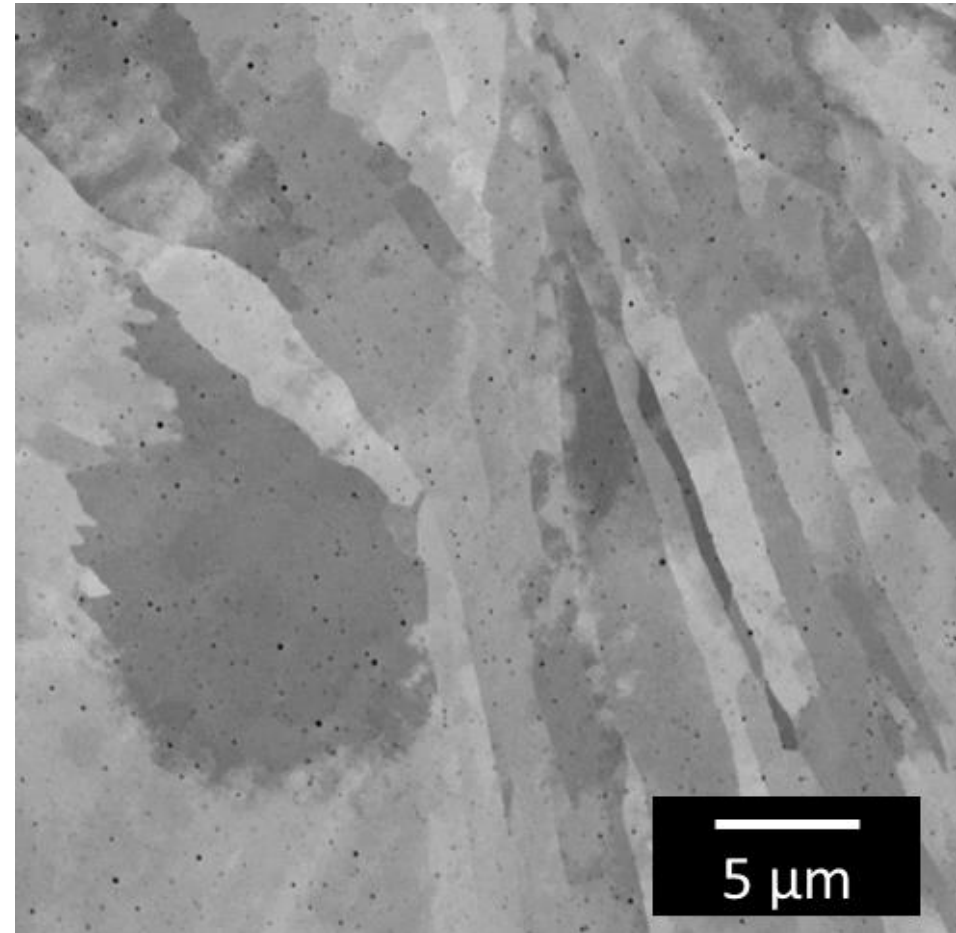
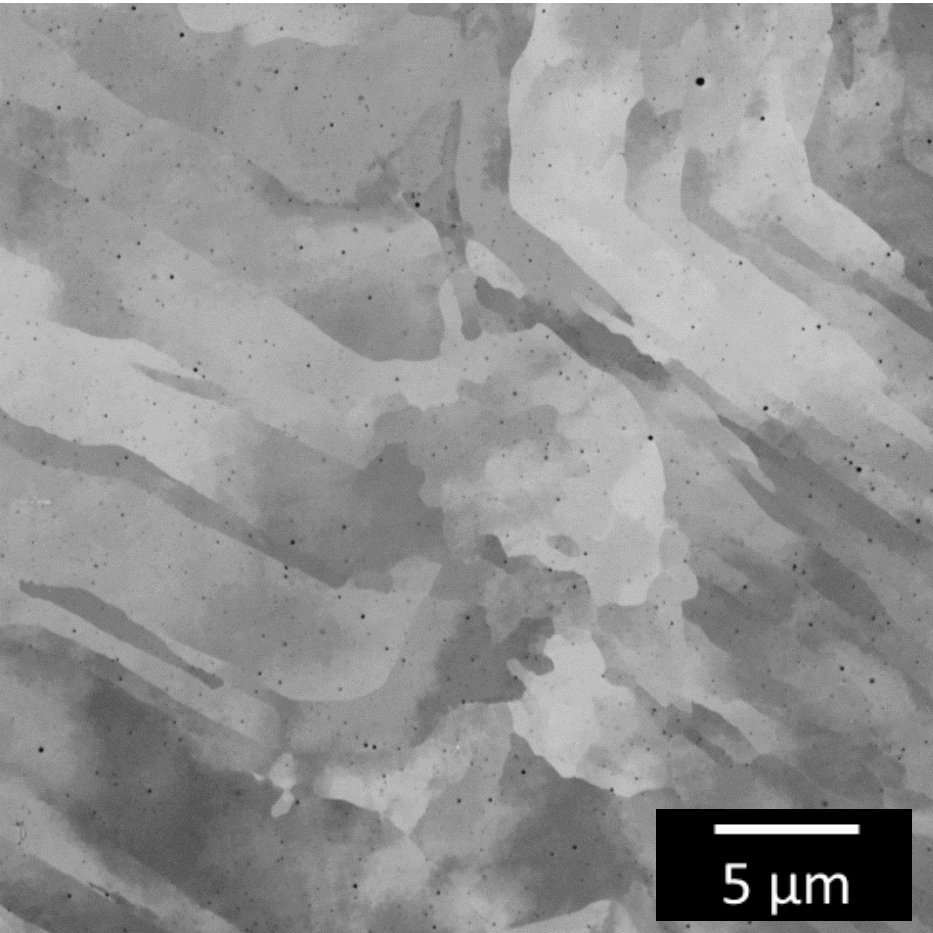
- The advanced dispersion coating (ADC) technique did not deform the metallic powder.
- The ADC technique fully coats the metallic powders with nano-scale oxides
- Both uncoated and coated powders qualitatively passed the Hall flow test.
- The technique does not affect the printability of the powder lot.

# Leveraging L-PBF to Produce Oxide Dispersion Strengthened Alloys



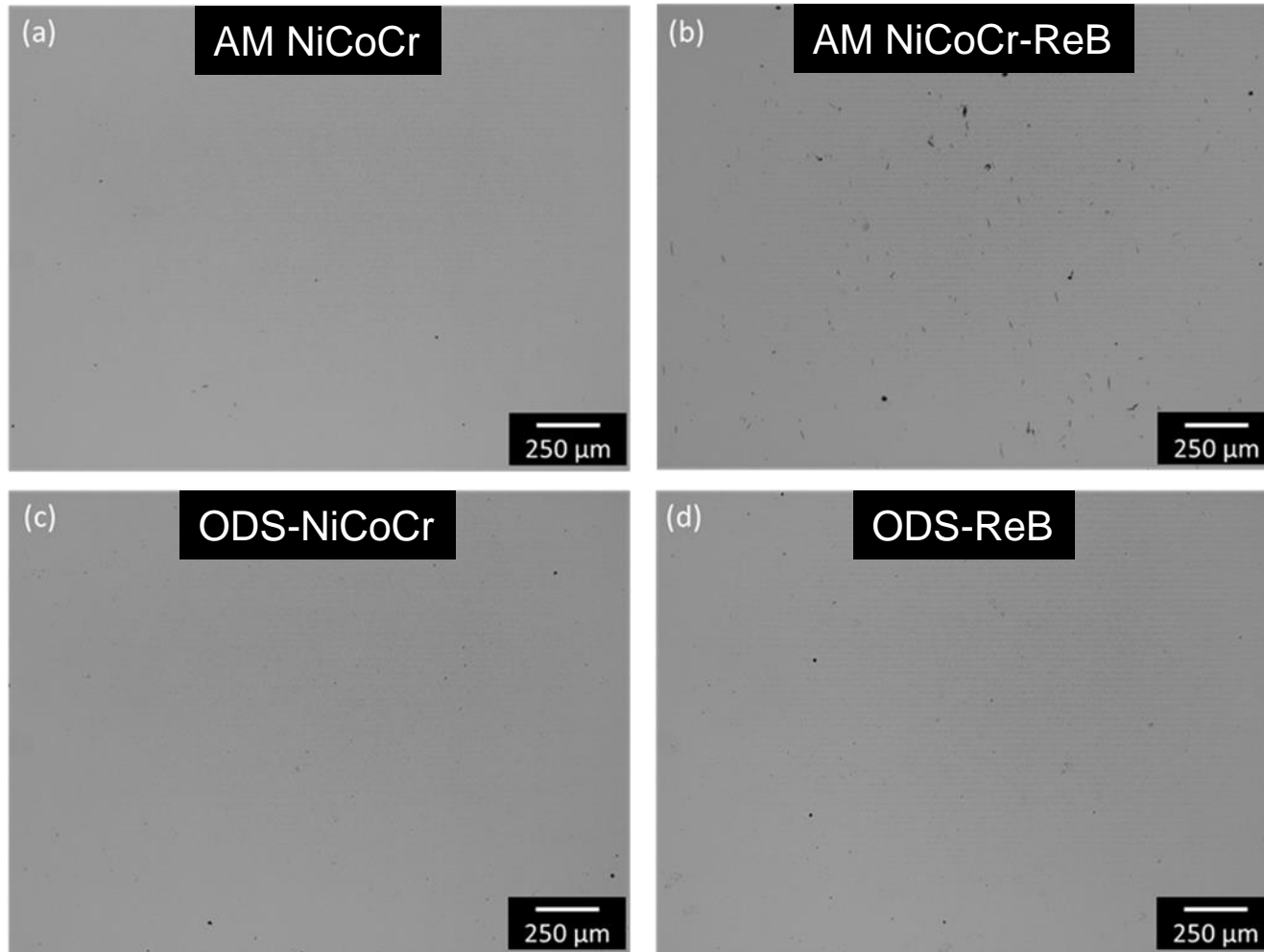
L-PBF successfully disperses the nano-scale Y<sub>2</sub>O<sub>3</sub> particles throughout the AM build

# ODS-NiCoCr Microstructure



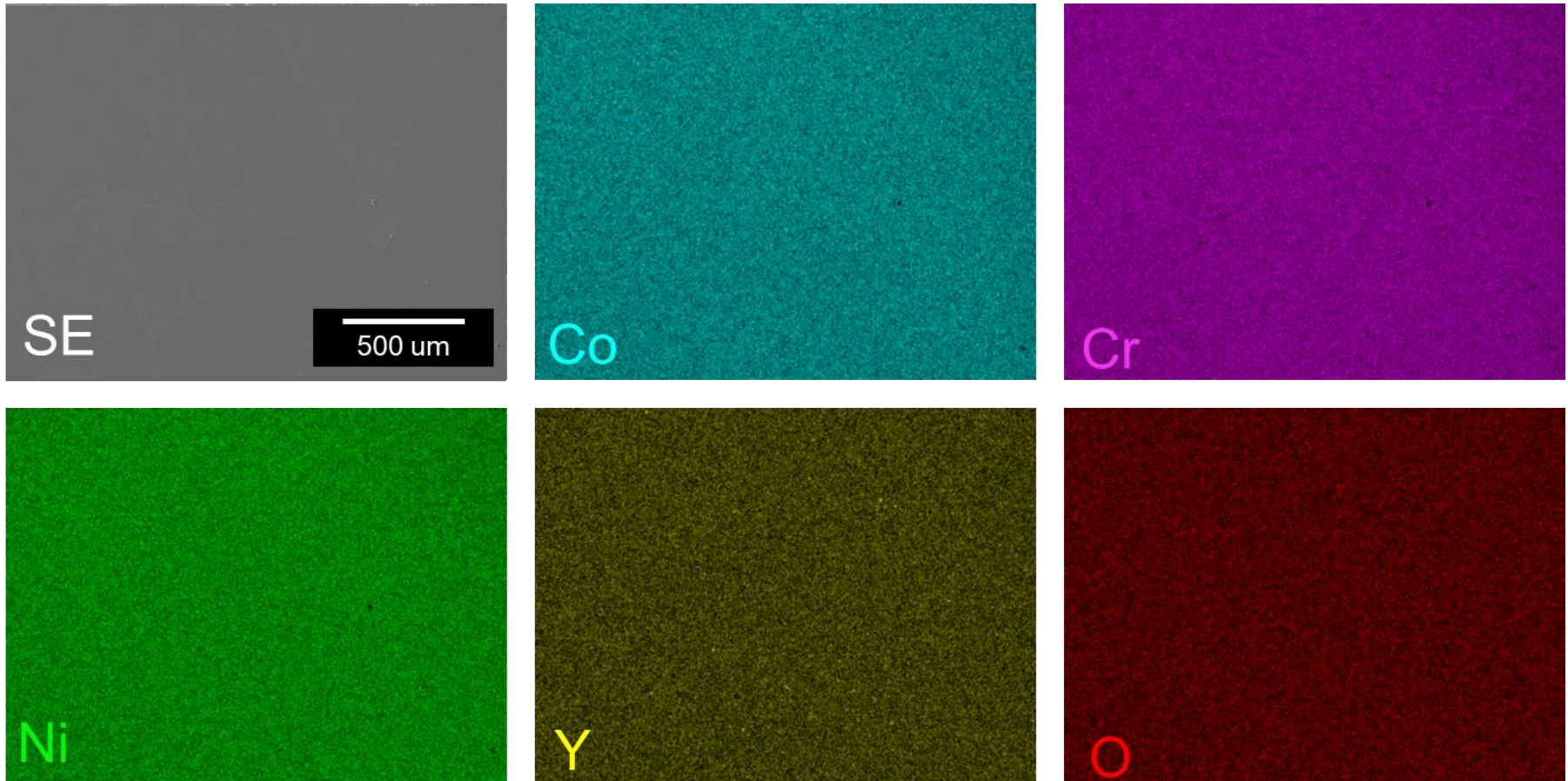
Nano-scale  $\text{Y}_2\text{O}_3$  particles are randomly dispersed throughout microstructure.

# Microstructures - Porosity



99.9% dense parts were successfully built for AM NiCoCr, ODS-NiCoCr, and ODS-ReB powder lots. Uncoated AM NiCoCr-ReB powder exhibited extensive micro-cracking. Result suggests coated powder is more printable.

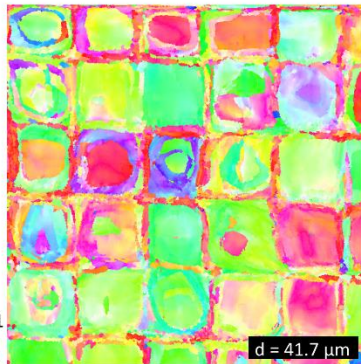
# EDS – NiCoCr-ODS Microstructure



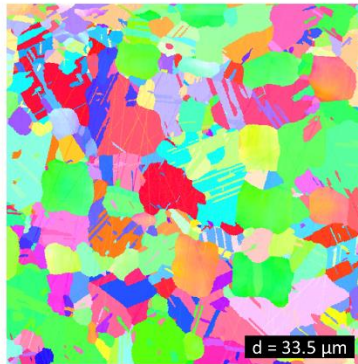
- Large ( $>20\mu\text{m}$ )  $\text{Y}_2\text{O}_3$  particles are not present in AM builds
- NiCoCr matrix remained a random solid solution during L-PBF process.

# Microstructure Analysis

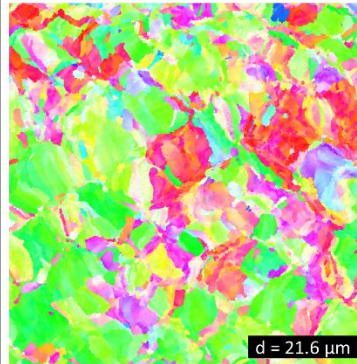
NiCoCr As-Built



NiCoCr HIP



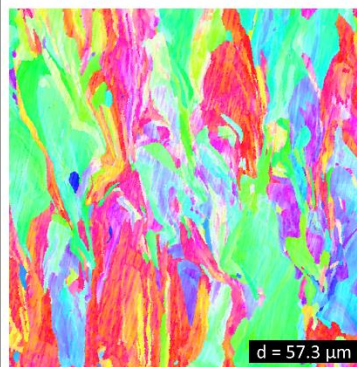
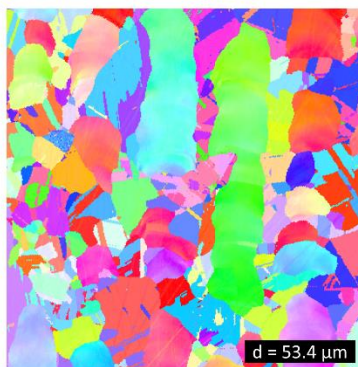
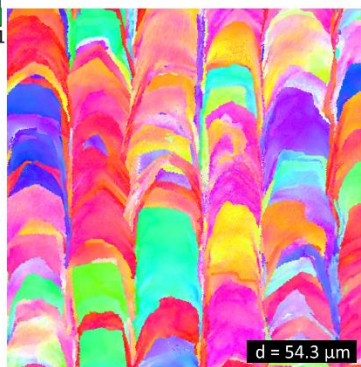
Representative ODS



- $Y_2O_3$  particles have pinned the grain boundaries in the ODS AM builds.
- The HIP cycle successfully removed residual stresses for AM samples



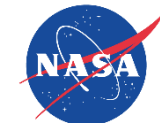
IPF

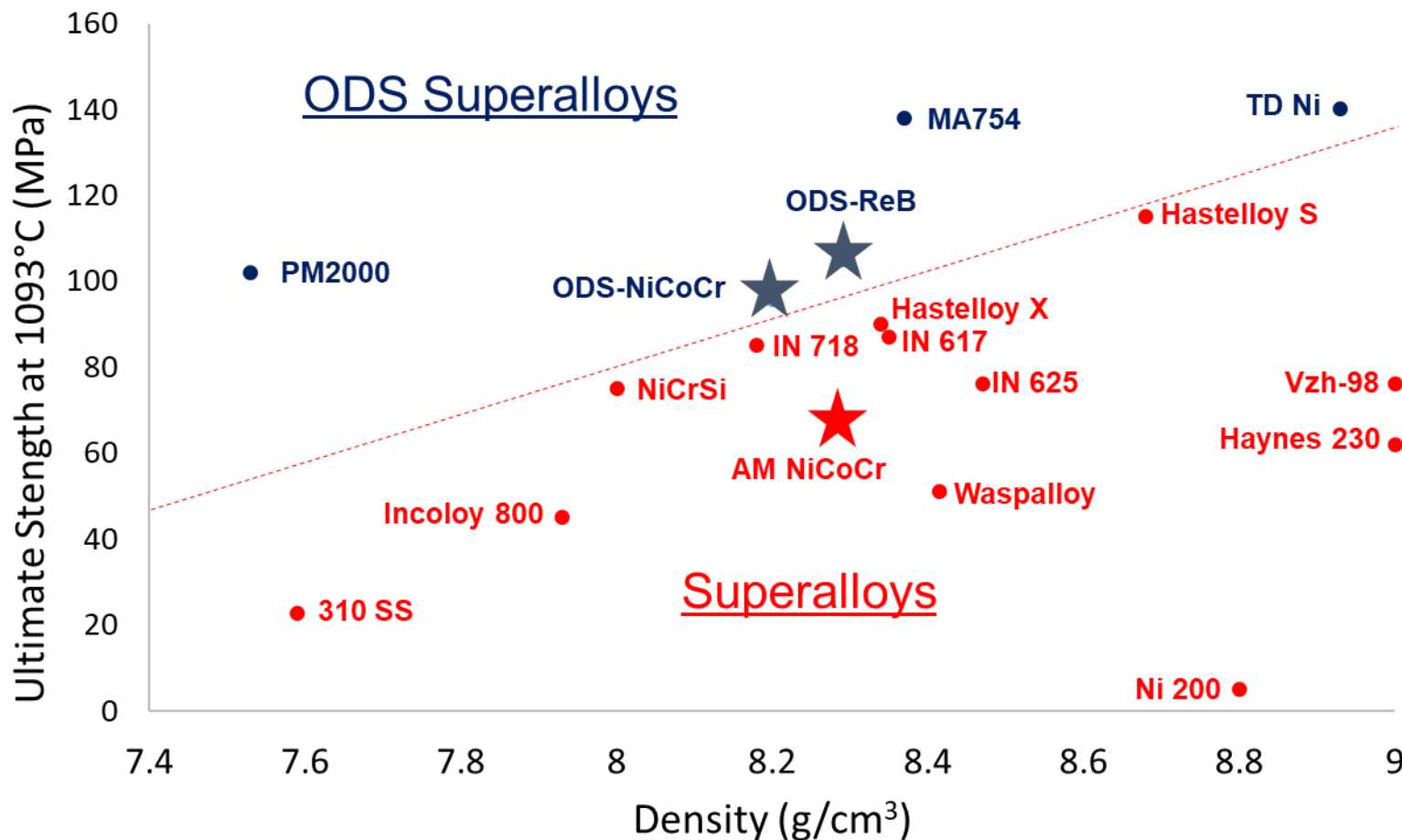
100 μm

## Residual Stress

Alloy	As-Built – Build direction	As-Built – 90° from build direction	HIP – Build direction	HIP – 90° from Built direction
AM NiCoCr	$34 \pm 35$	$141 \pm 96$	$-5 \pm 3$	$-4 \pm 6$
ODS-NiCoCr	$320 \pm 51$	$185 \pm 49$	$-11 \pm 9$	$-12 \pm 9$
ODS-ReB	$321 \pm 52$	$179 \pm 47$	$-8 \pm 5$	$-12 \pm 6$



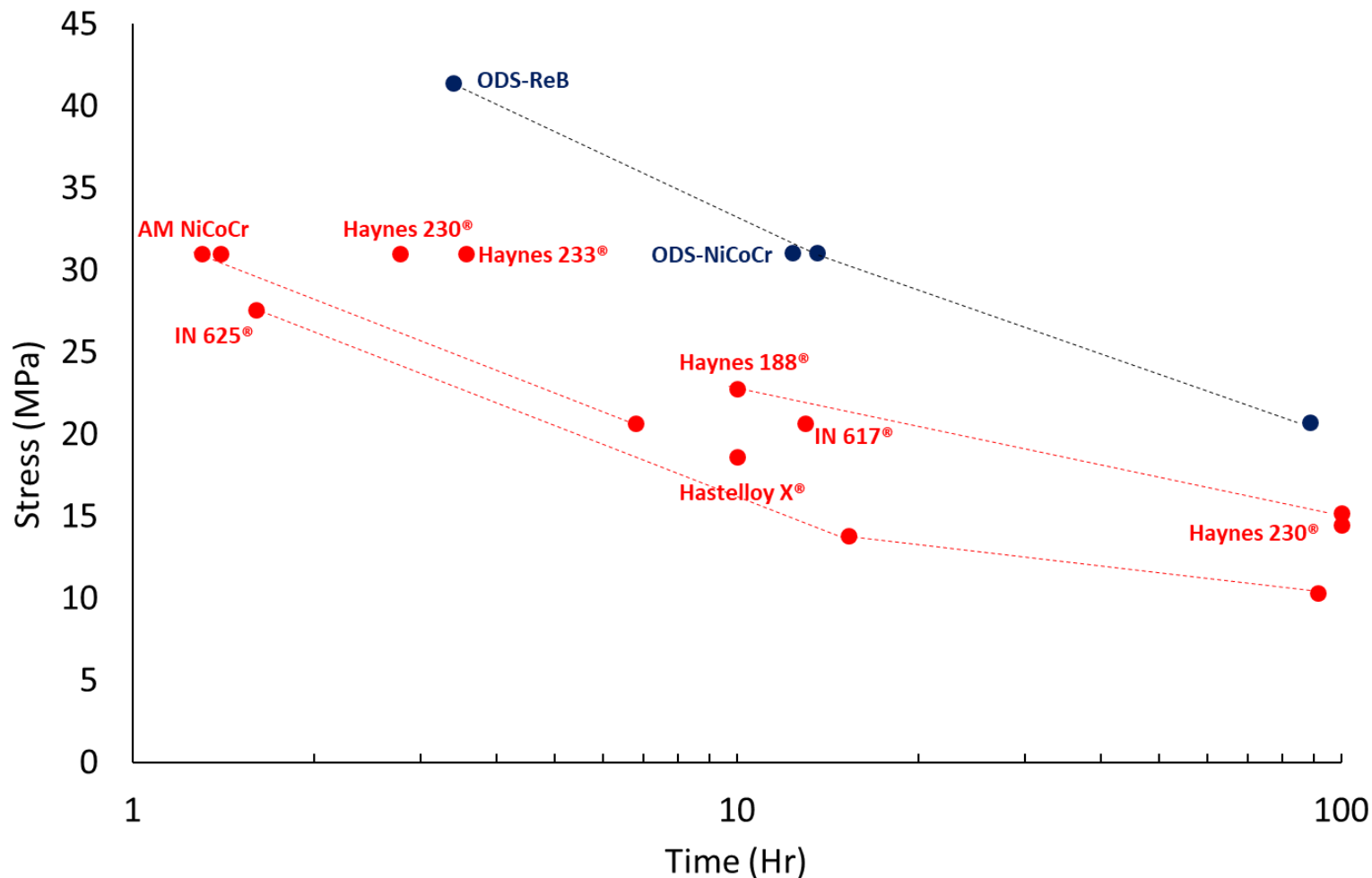
# Tensile Strength vs Density Comparison



Scatter plot confirms the successful production of a ODS alloy using AM



# Creep Rupture Lives Comparison- 1093°C



Scatter plot confirms the successful production of a ODS alloy using AM

# Oxide Dispersion Strengthened MPEA Combustor Dome



# LEW-19886-1: “Additively Manufactured Oxide Dispersion Strengthened Medium Entropy Alloys for High Temperature Applications”

## Model Driven MPEA Design

### Goals to improve on previous NiCoCr Entropy Alloy:

- 1.) Maximize solid solution strengthening
- 2.) Maintain solid solution matrix
- 3.) Add grain boundary carbides
- 4.) Reduce freezing range to under 100°C for printability

**Over 50 simulations provided an optimized composition named Alloy X**

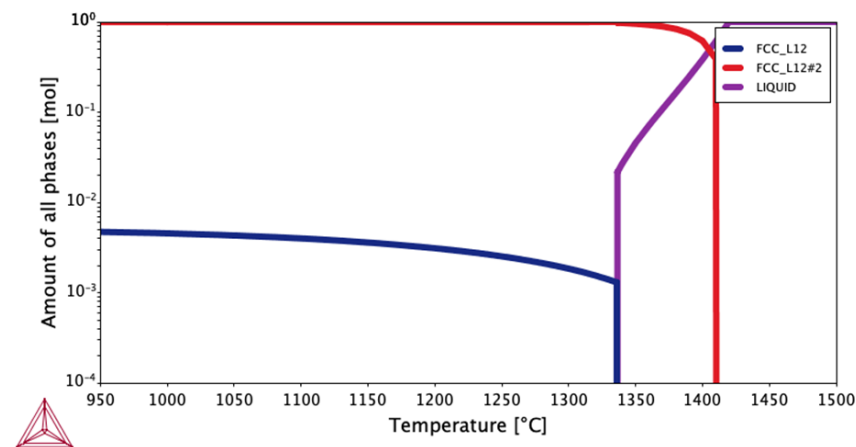
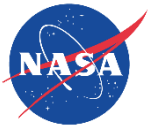


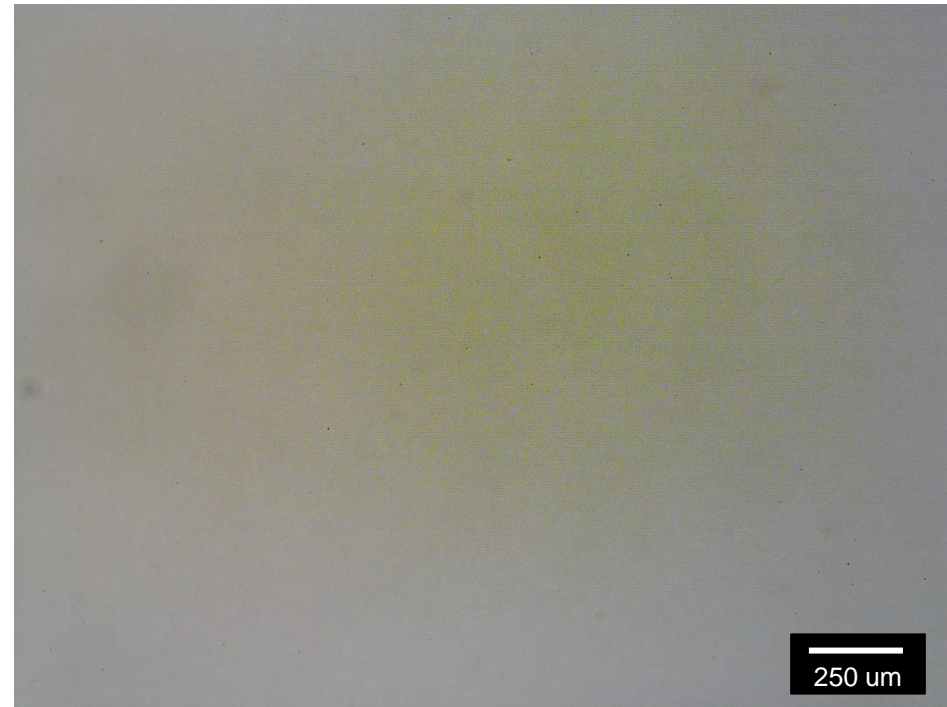
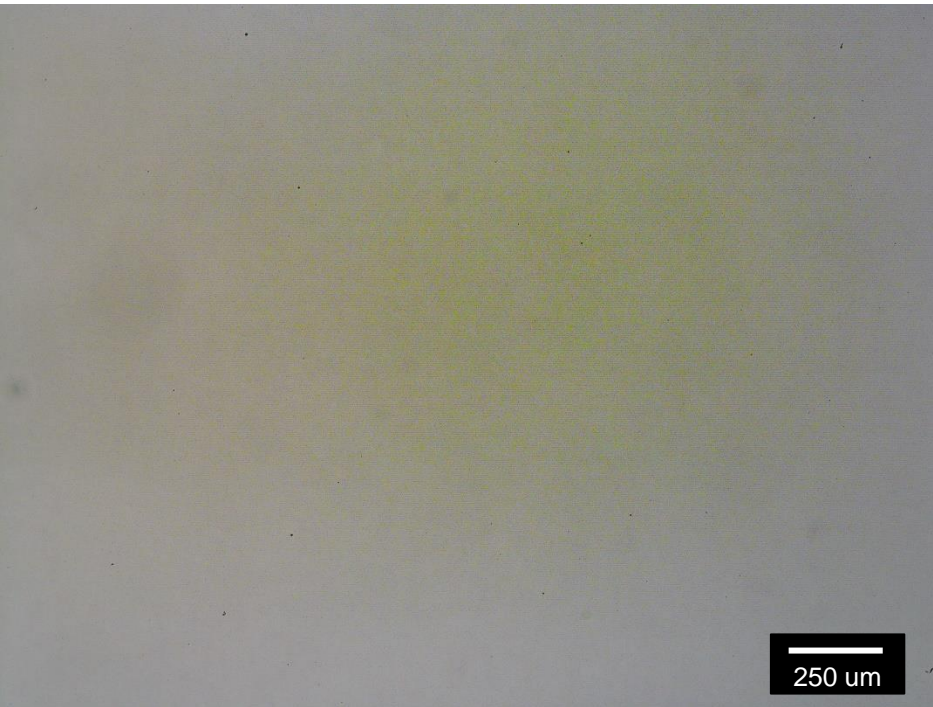
Figure: CALPHAD simulation of phase formation in new composition. No intermetallic or TCP phases are predicted.

Models calculated by C. Kantzos

	Ni	Co	Cr	Re	Al	Ti	Nb	Mo	W	Zr	C	B
Old Composition	Bal.	32	30	1.5								.003
New Composition (Alloy X)	Bal.	33	29	1.5	x	x	x	x	x	x	x	x



# Optical Microscopy – Alloy X - ODS HIPed

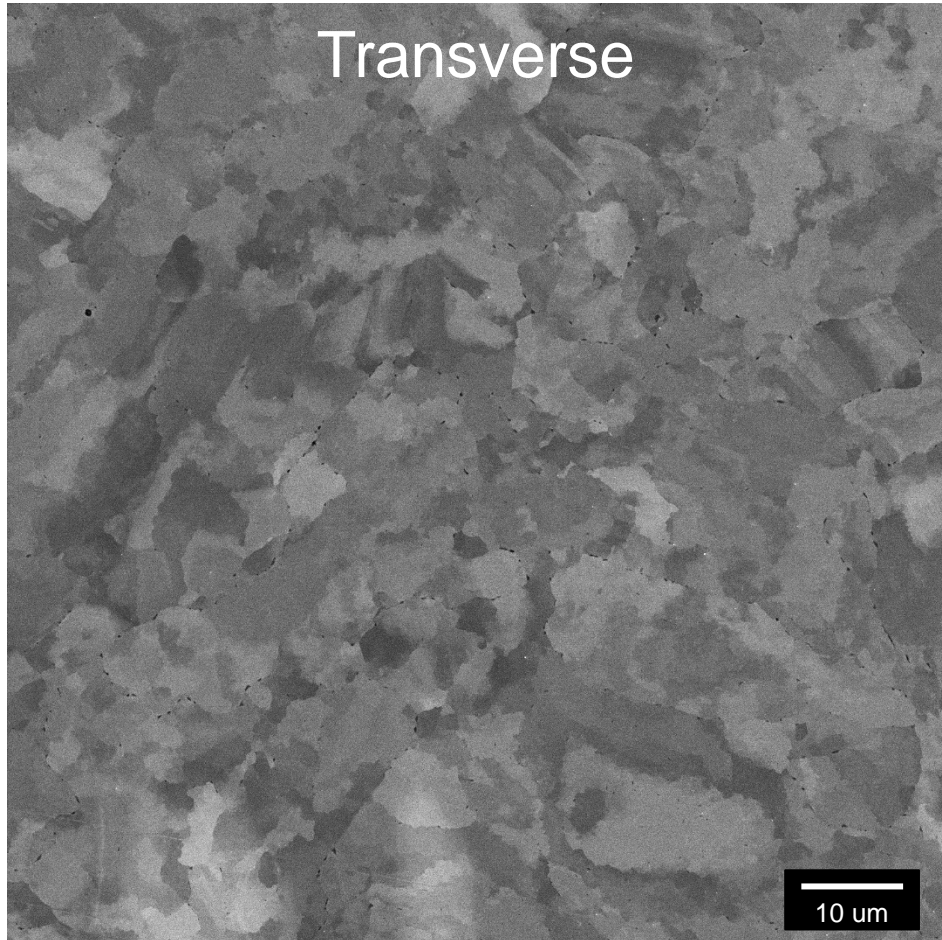


HIP removes most defects.

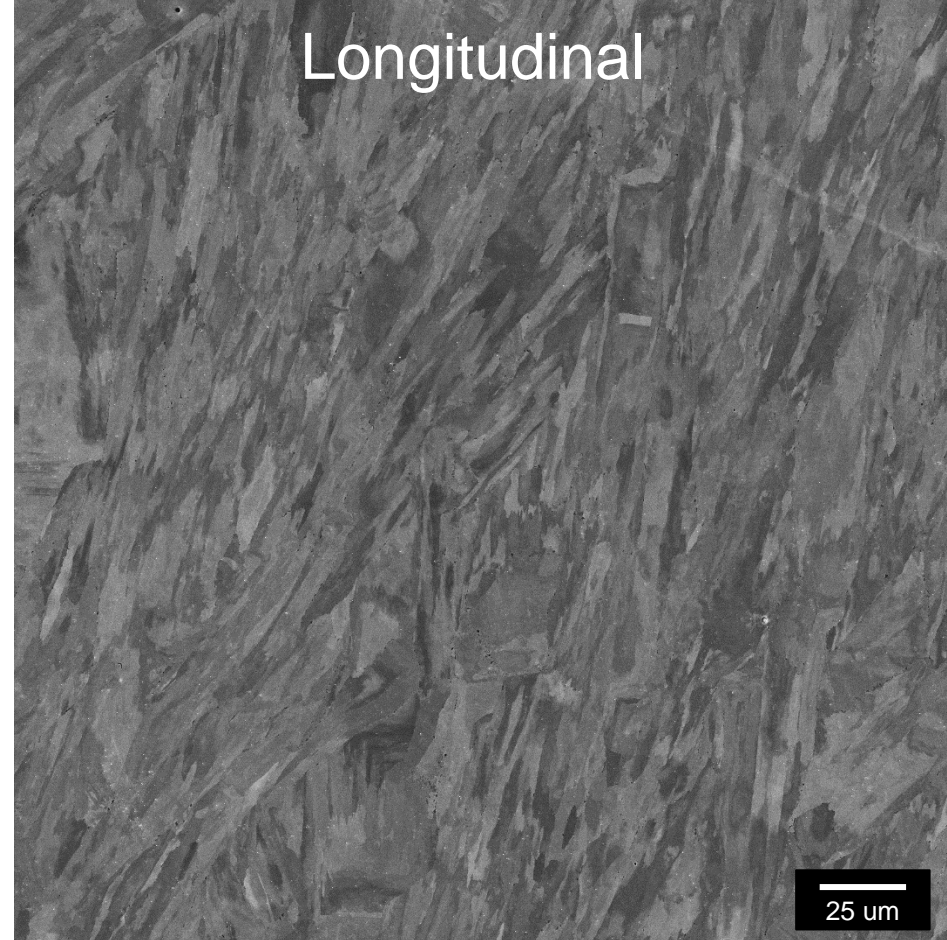
Average 99.9912% density

# SEM – Alloy X - ODS HIPed

Transverse

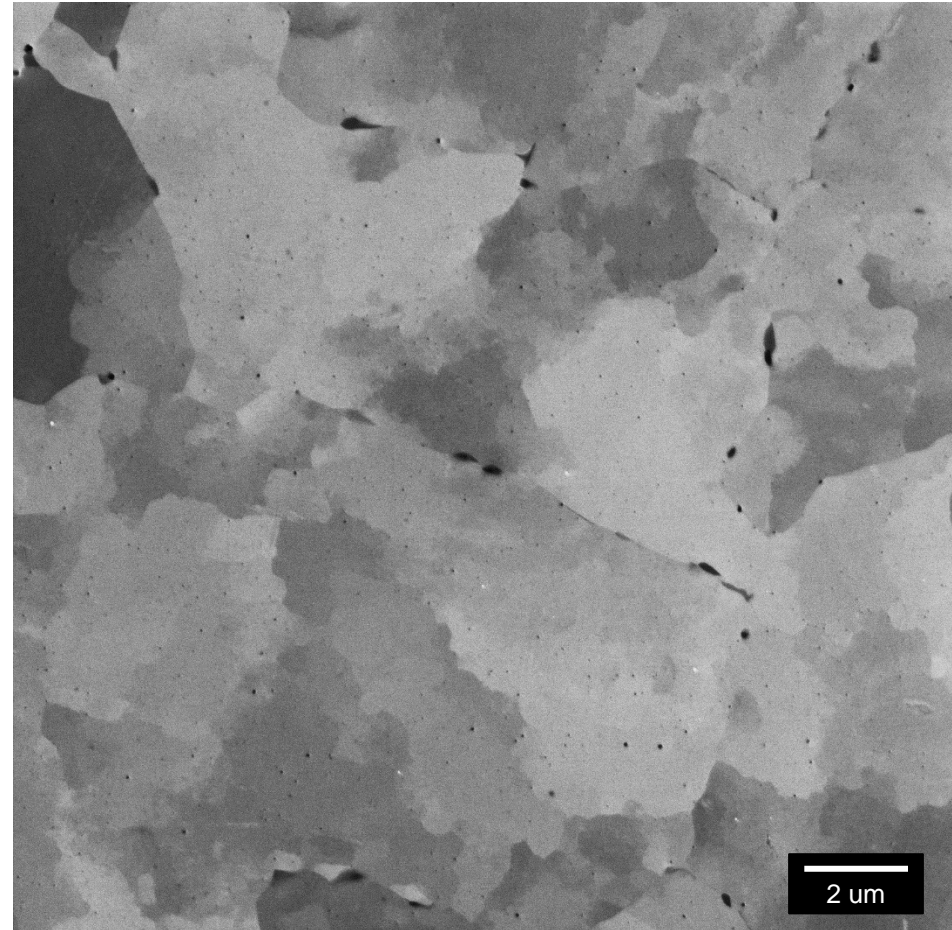
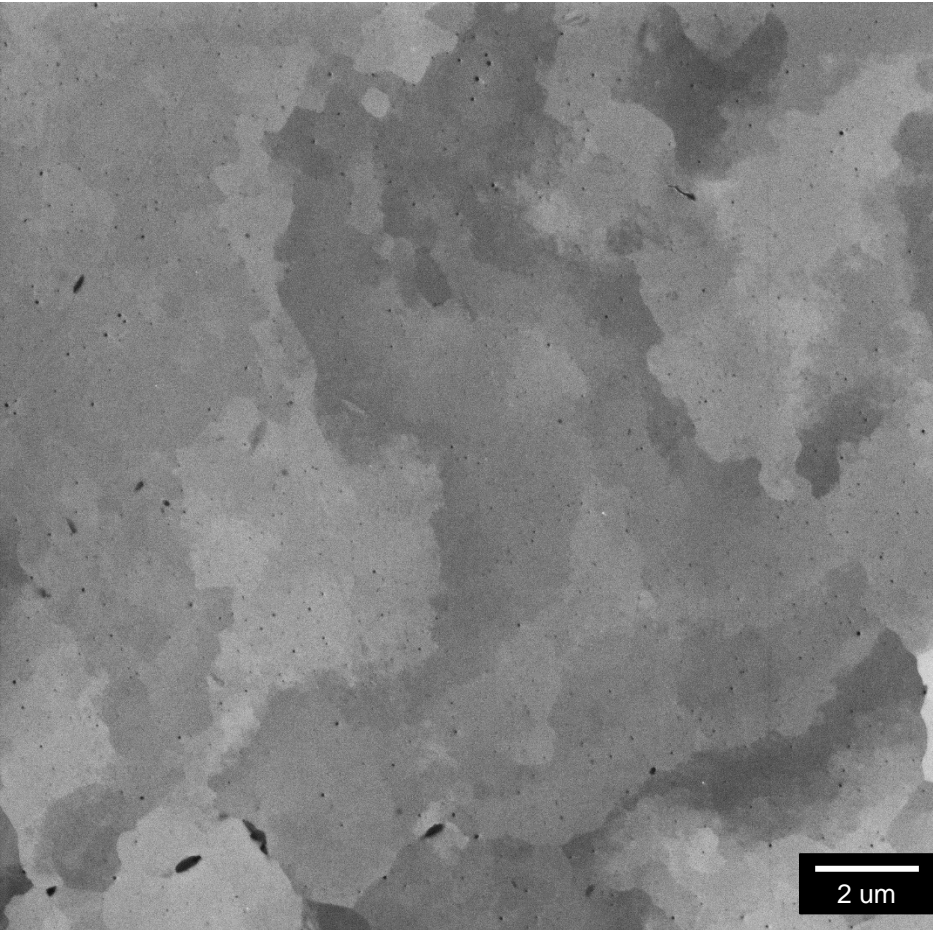


Longitudinal



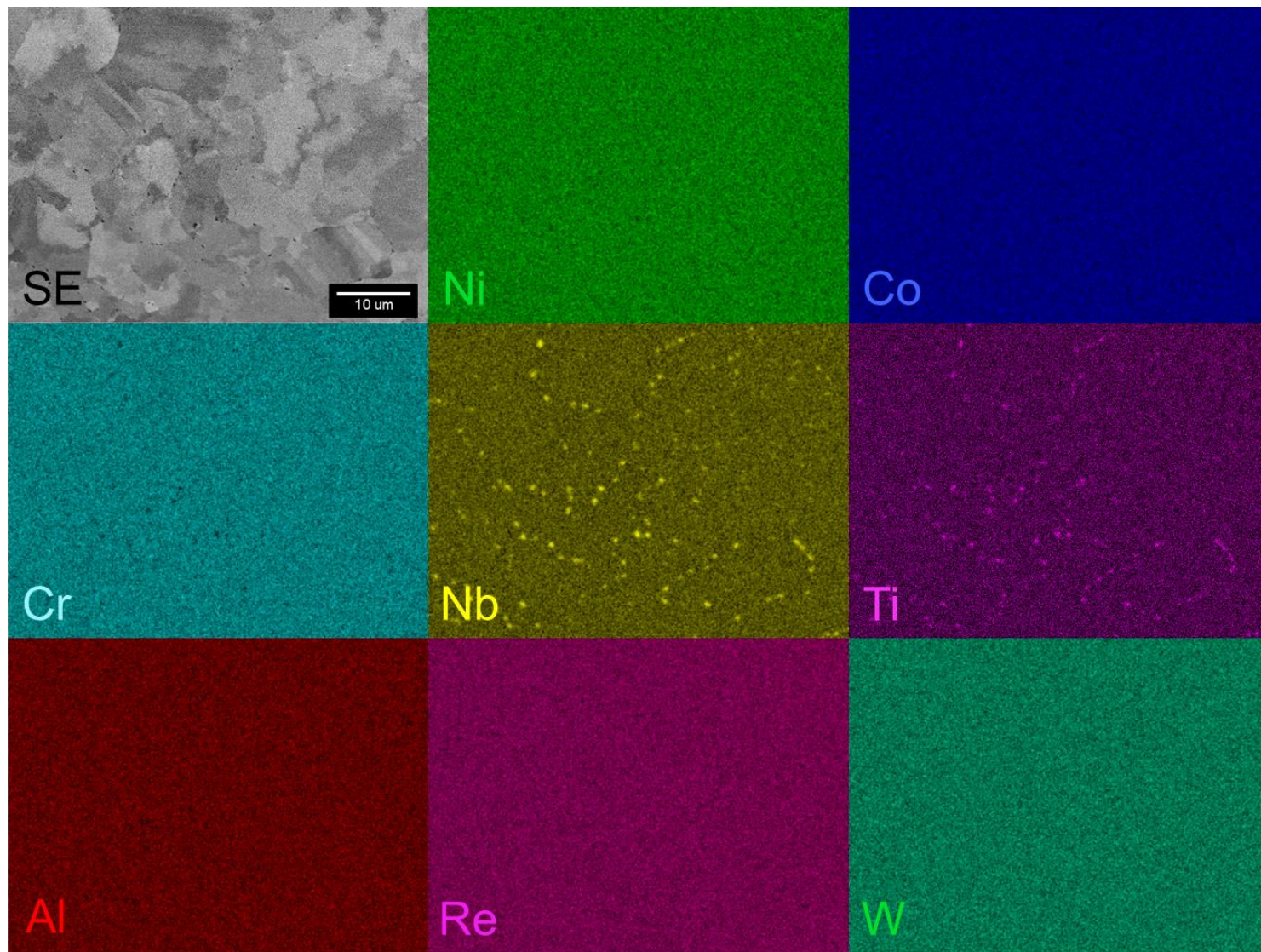
Low magnification SEM images reveal anisotropic grain structure. Suggests oxides are present pinning grain boundaries during HIP

# SEM – Alloy X - ODS HIPed

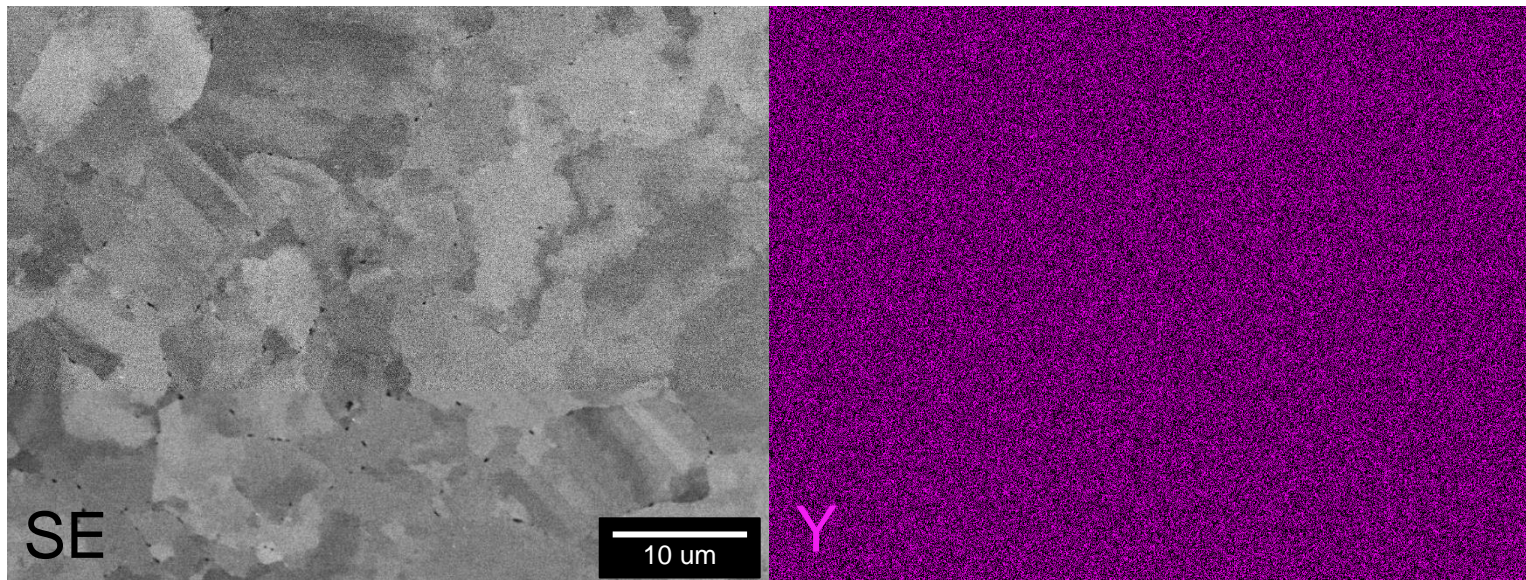


High resolution SEM reveals dispersed nano-oxides and grain boundary phases.

# SEM-EDS – Alloy X - ODS HIPed



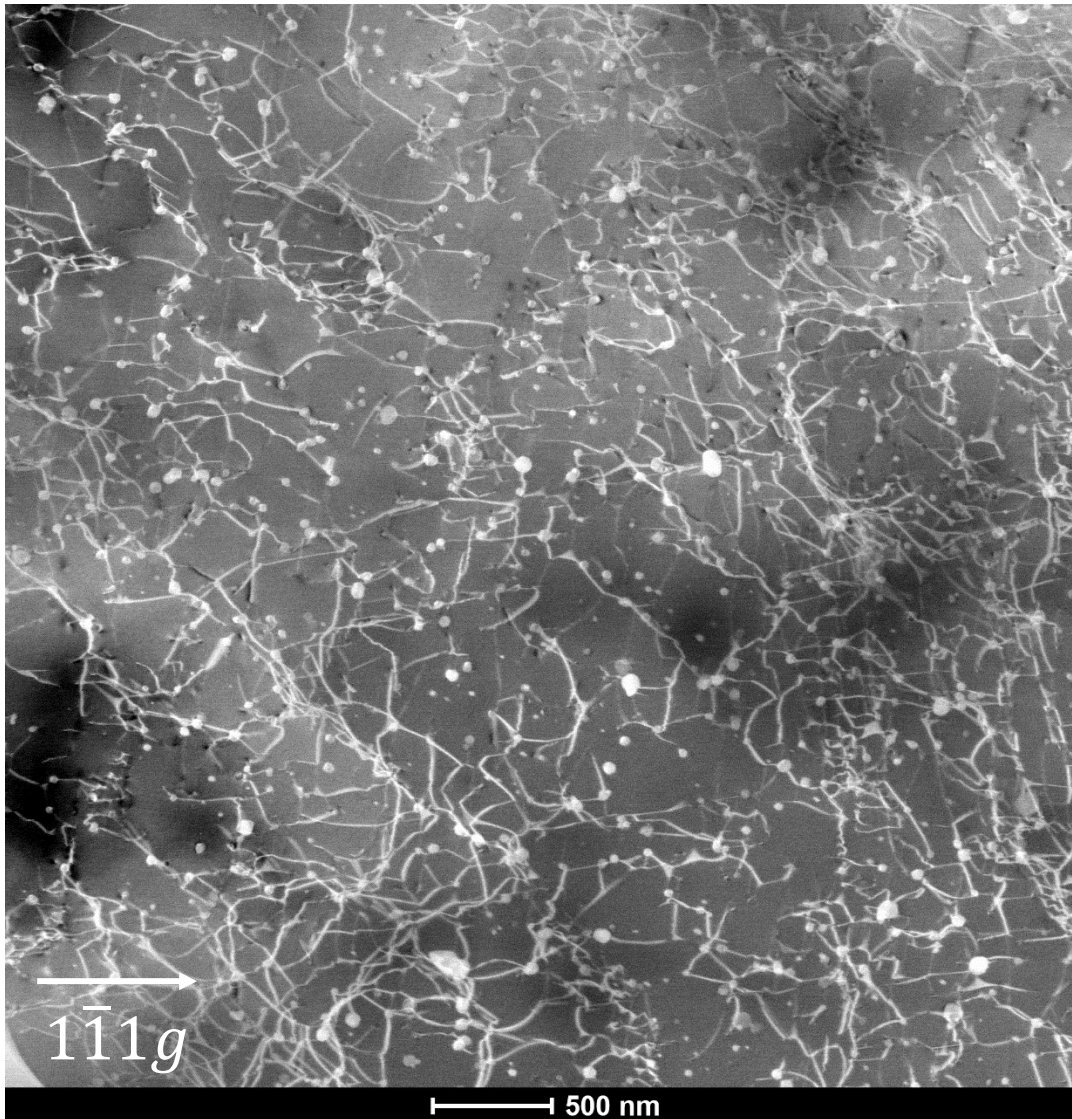
# SEM-EDS – Alloy X - ODS HIPed



- SEM-EDS confirms the presence of Nb- and Ti-rich carbides along grain boundaries.
- SEM-EDS confirms the new composition has maintained a solid solution matrix.
- No intermetallic or TCP phases were observed.
- Bulk  $Y_2O_3$  appears to have been avoided as well.

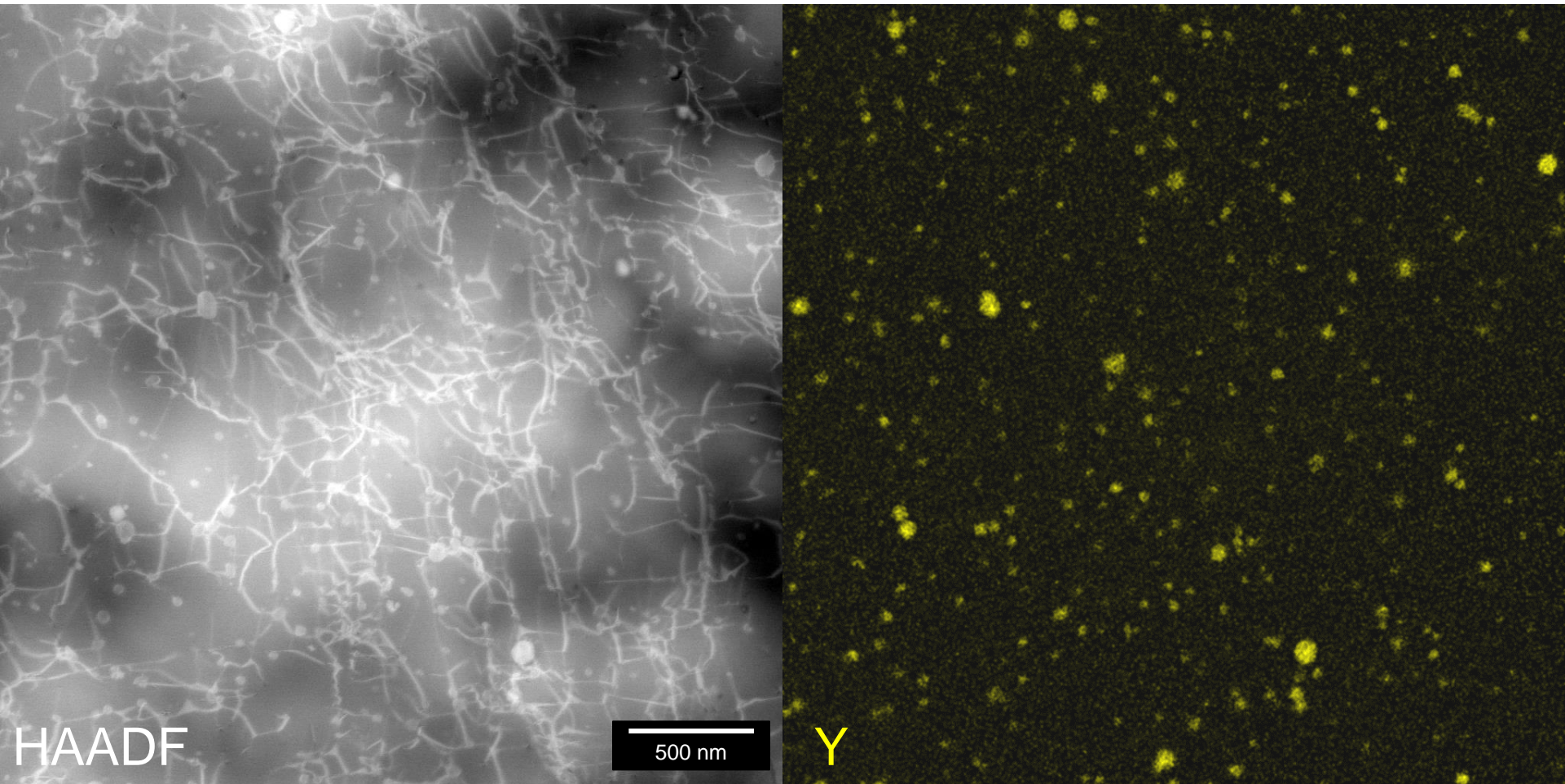
## Confirmation of CALPHAD model predictions

# STEM-EDS Analysis

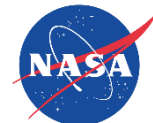


HAADF STEM analysis confirms presence of nano dispersoids within Alloy X ODS matrix. Highly dense dislocation networks still present after HIP step due to dislocation pinning by dispersoids.

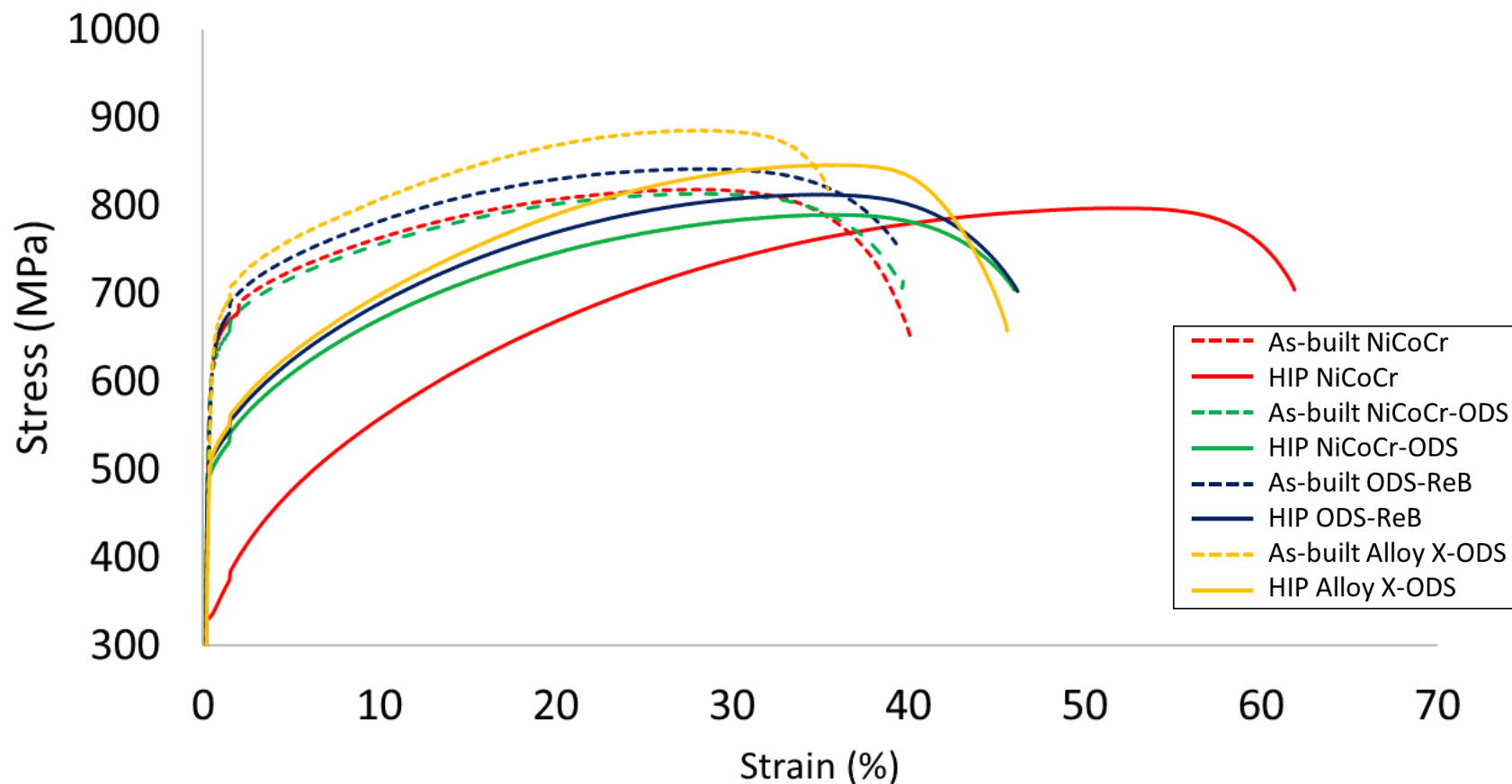
# STEM-EDS



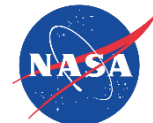
STEM-EDS analysis confirms high density of nano-scale  $\text{Y}_2\text{O}_3$  particles throughout bulk. No oxide agglomeration was present. Most other elements did not react with oxides (Nb and Ti were exceptions).



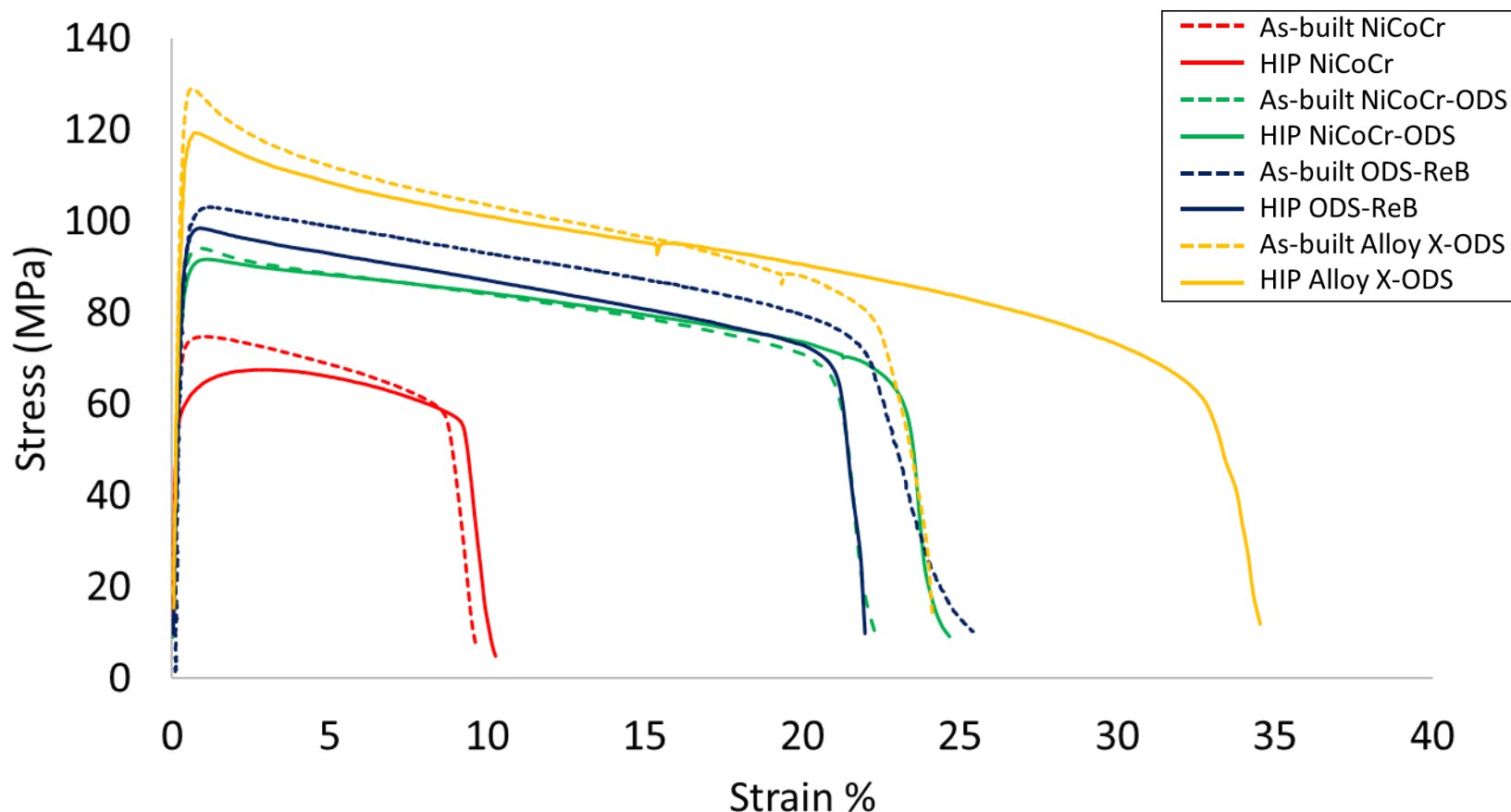
# Mechanical Results – Room Temperature Tensile



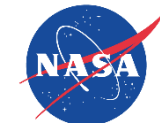
- **X-ODS presents higher ultimate tensile strength at room temperature while maintaining the MPEAs fundamental high ductility.**



# Mechanical Results – 1093°C Tensile

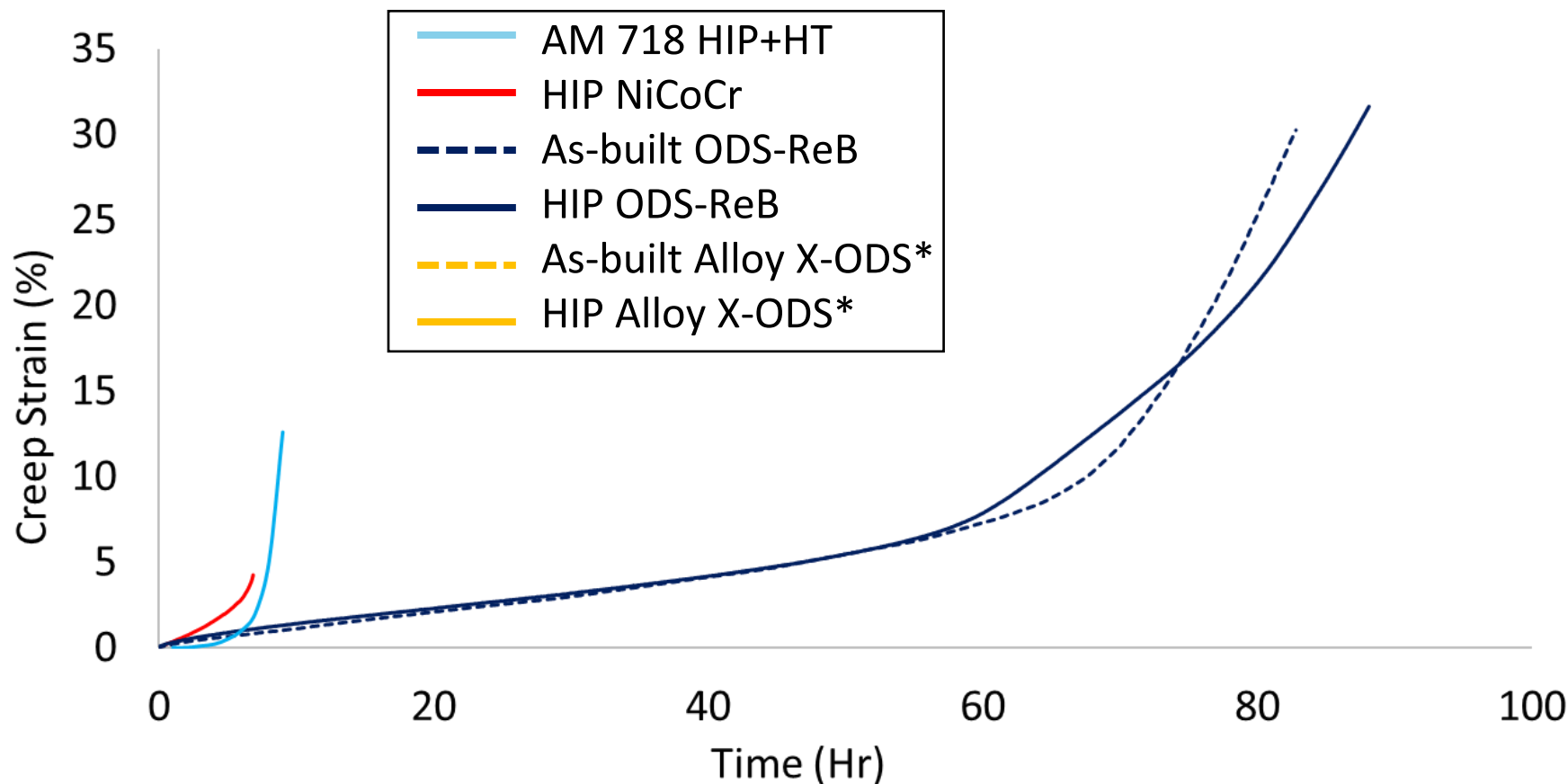


- **X-ODS provides almost double the tensile strength and over 3x the ductility compared to AM NiCoCr.**

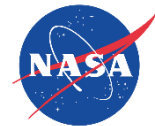


# Mechanical Results – 1093°C/20MPa

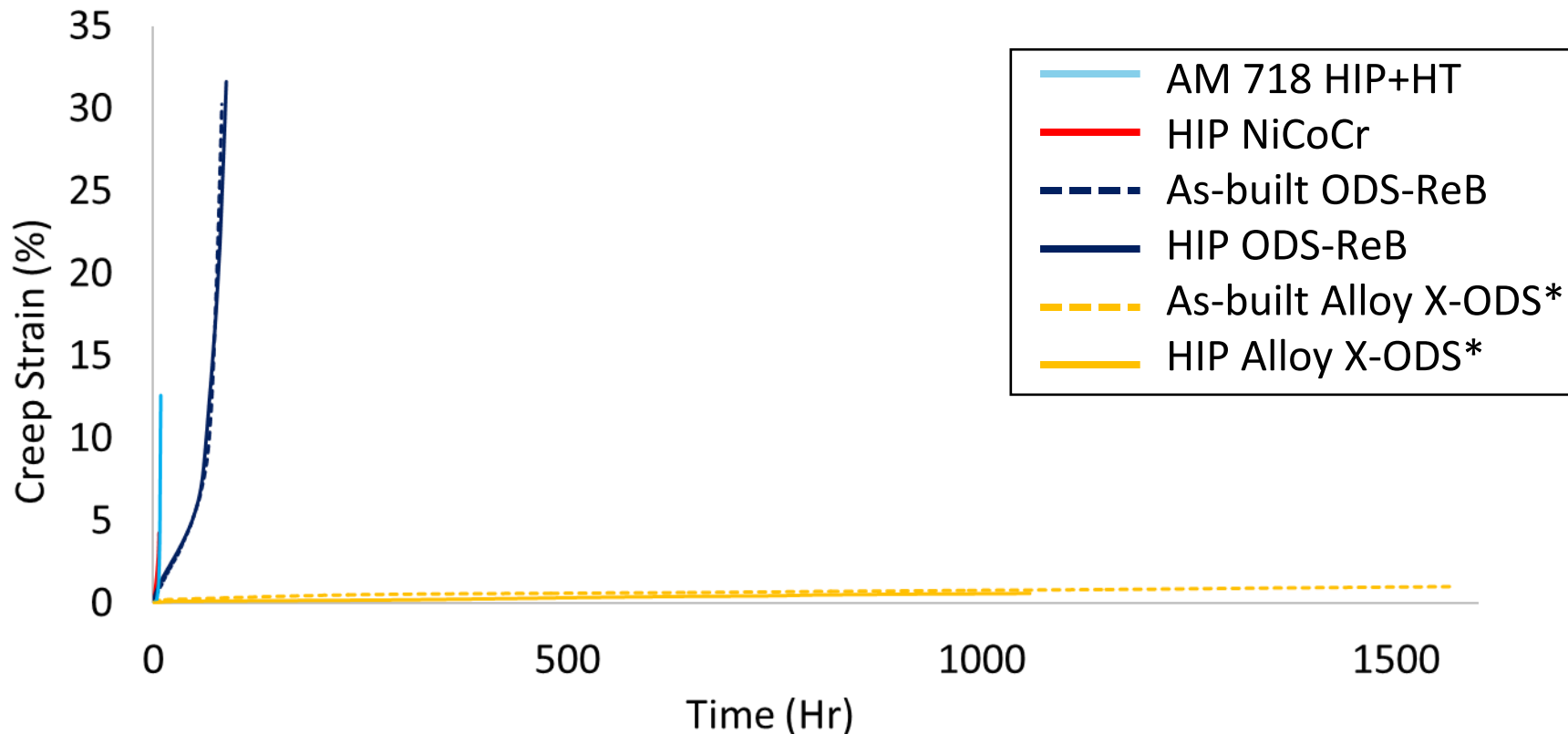
## Creep Rupture



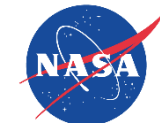
Incorporating oxides provides significant improvement in creep rupture life at 1093°C



# Mechanical Results – 1093°C/20MPa Creep Rupture

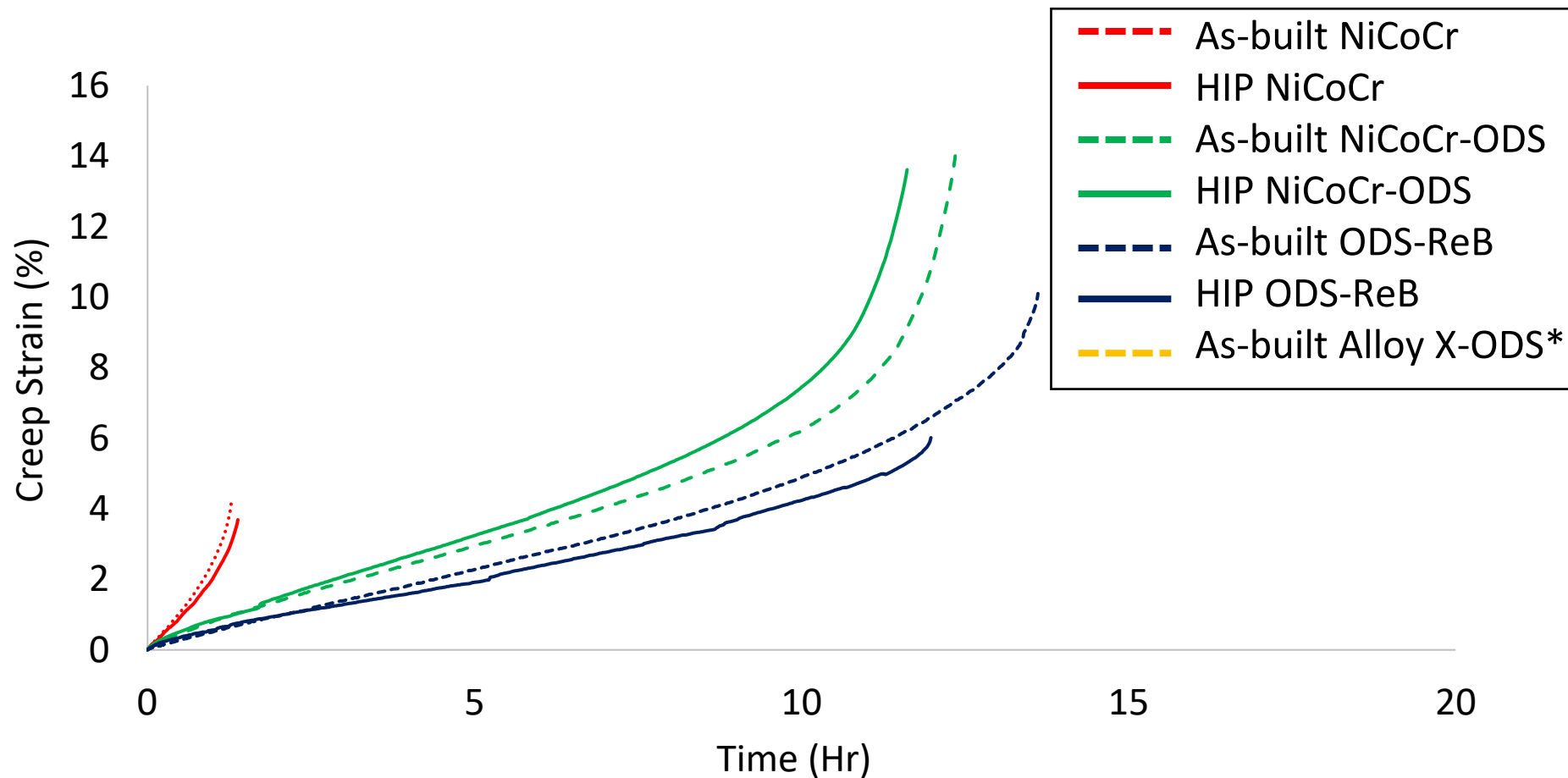


- **\*The as-built Alloy-X ODS test is still running ATM (1% strain at 1566 Hr).**
- **X-ODS is showing over a 225x improvement over NiCoCr in creep rupture life and almost a 75x improvement over superalloy 718.**
- **AM 718 built and tested by NASA's Henry DeGroh and Chris Kantzos**



# Mechanical Results – 1093°C/31MPa

## Creep Rupture

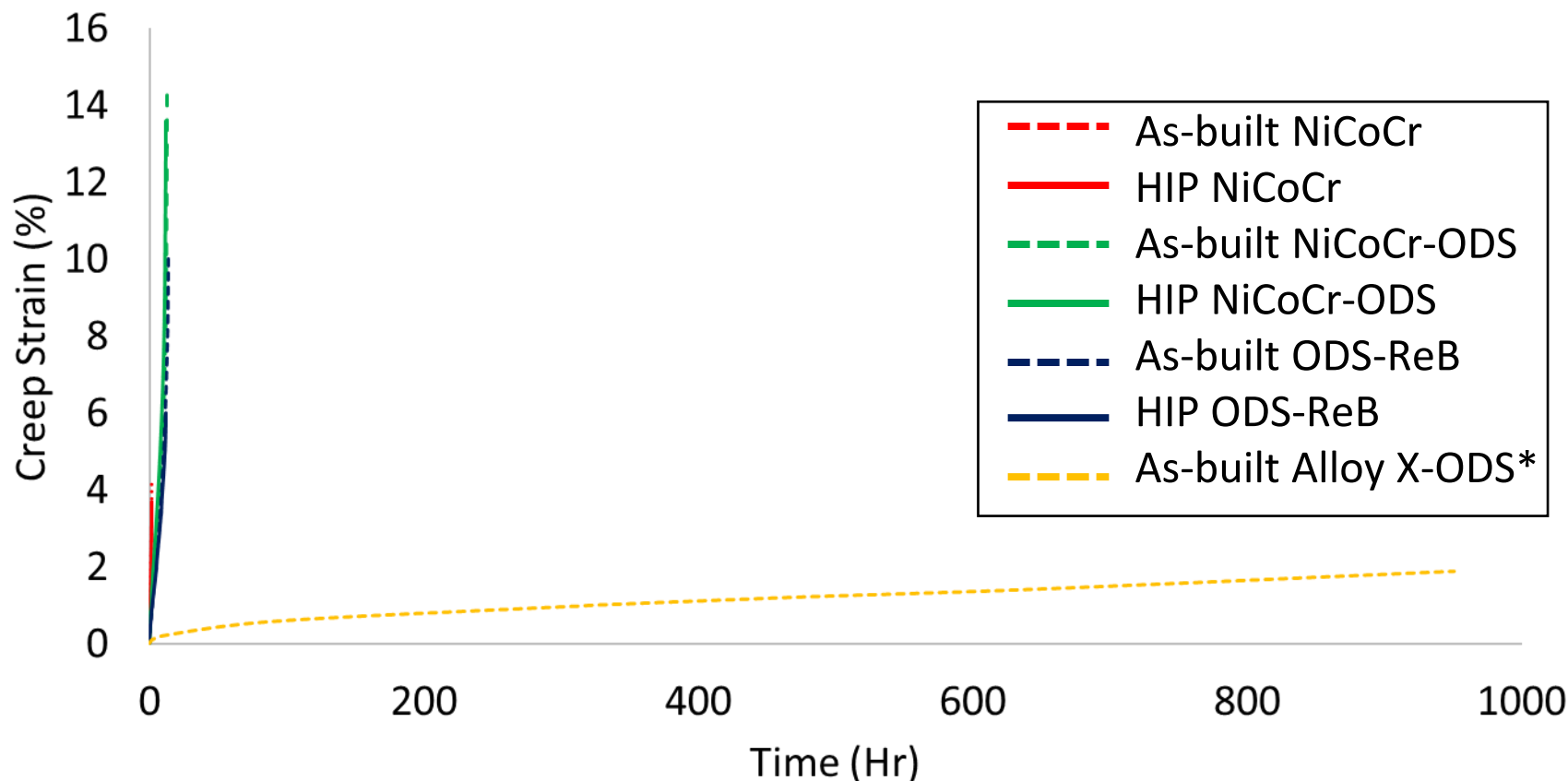


Incorporating oxides provides significant improvement in creep rupture life at 1093°C



# Mechanical Results – 1093°C/31MPa

## Creep Rupture

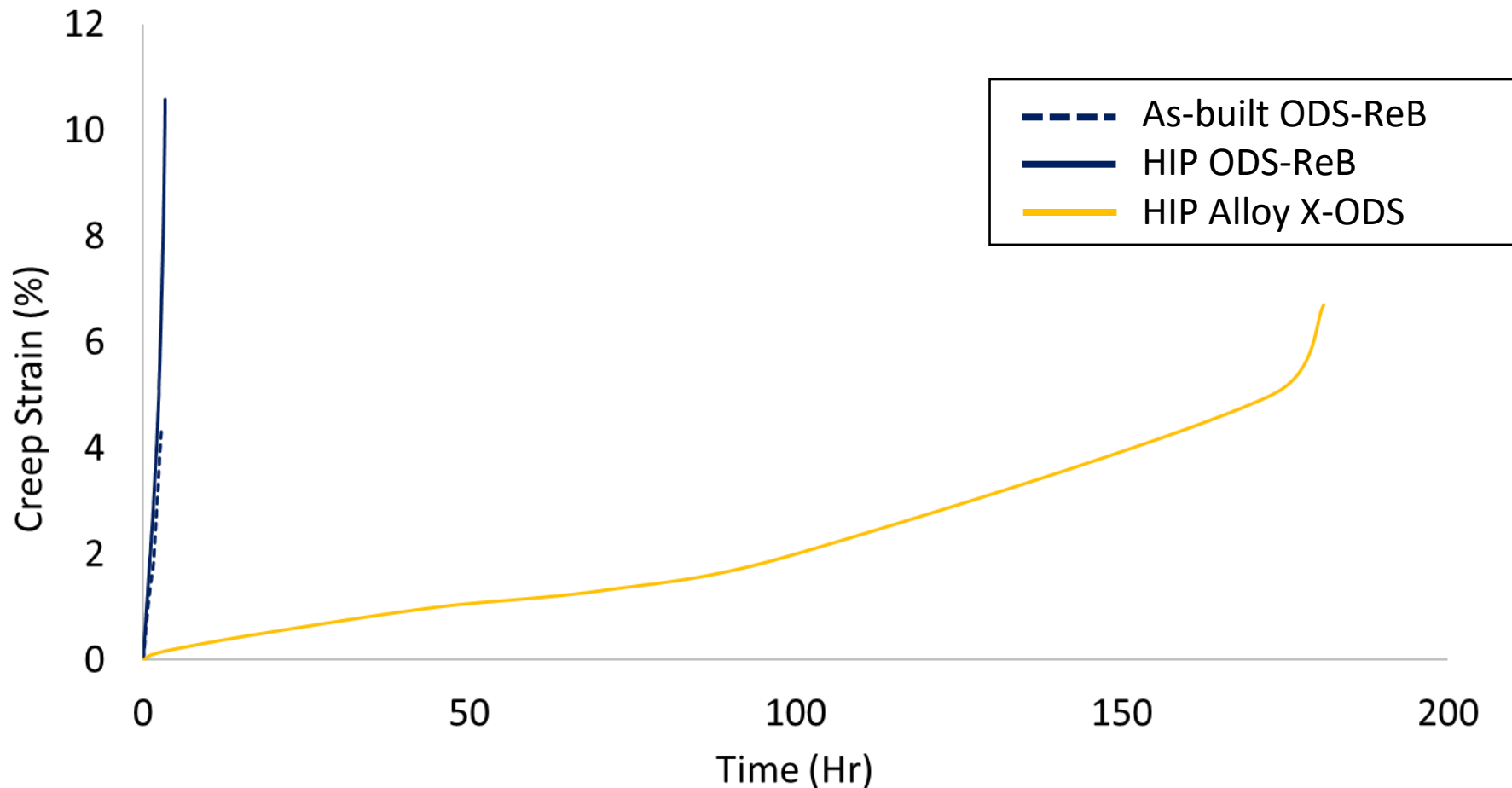


- **\*The as-built Alloy-X ODS test is still running ATM**
- **X-ODS is showing over a 750x improvement over NiCoCr in creep rupture life.**

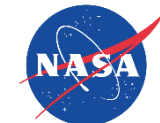


# Mechanical Results – 1093°C/41MPa

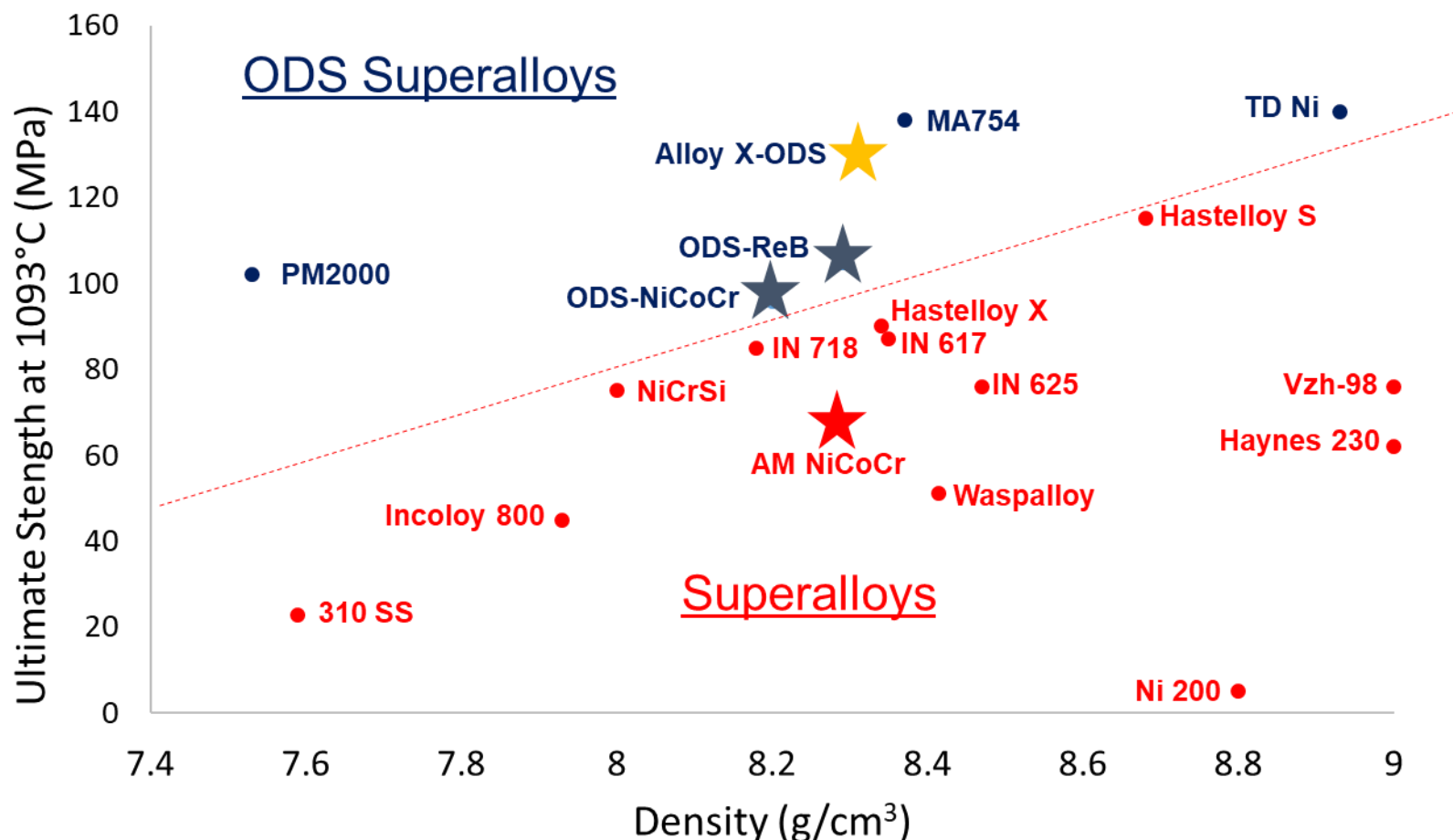
## Creep Rupture



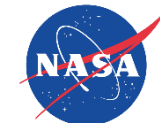
**X-ODS survived 50x longer than HIPed ODS-ReB during creep at this stress and temperature.**



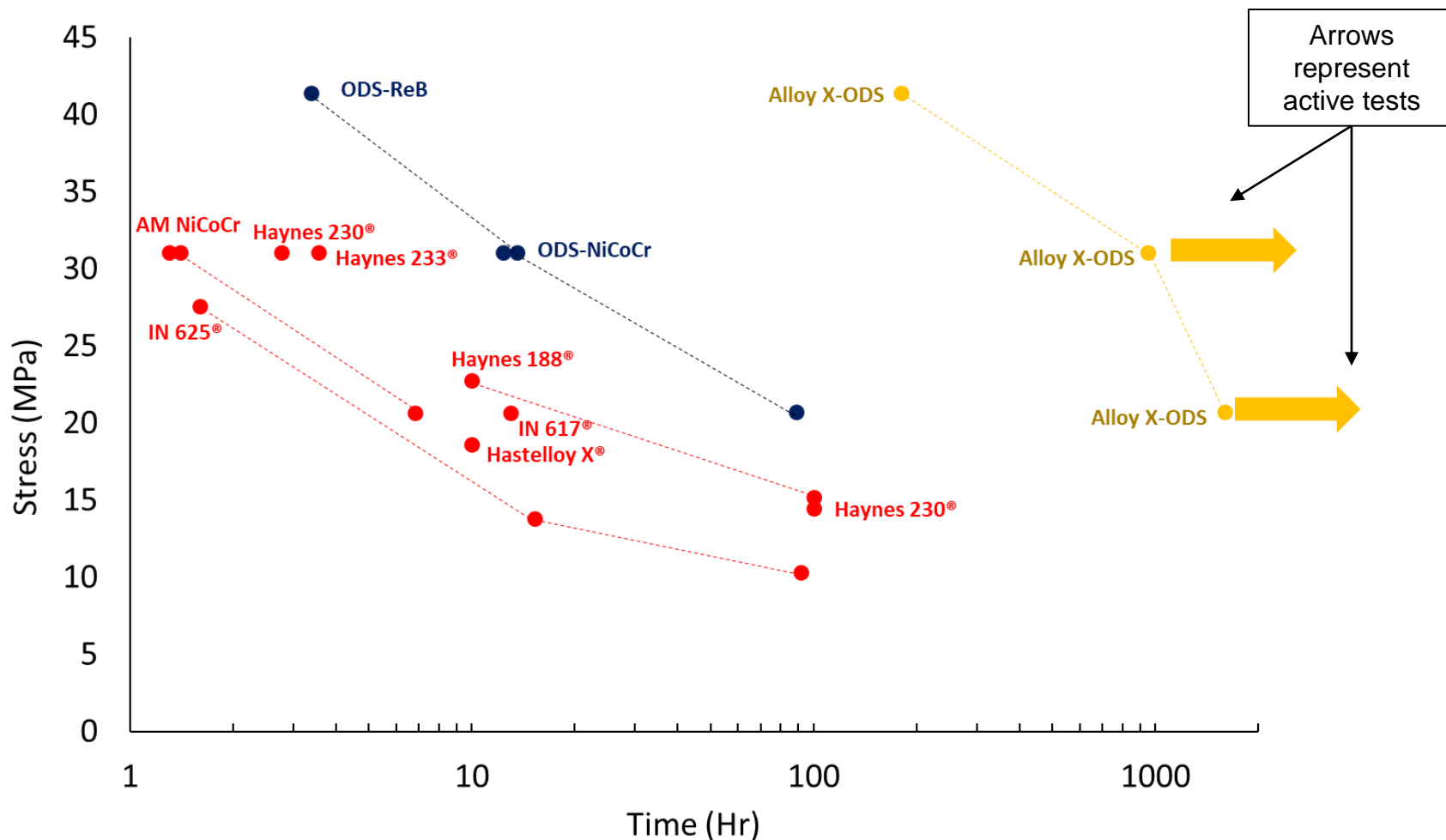
# Tensile Strength vs Density Comparison



Scatter plot confirms the successful production of a ODS alloy using AM



# Creep Rupture Lives Comparison- 1093°C



Even using the current unfinished creep data – Alloy X-ODS is revealing creep properties far superior to conventional superalloys at 1093°C.



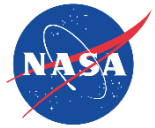
# Conclusions

## **LEW-20020-1: “Novel Fabrication Technique for Oxide Dispersion Strengthened (ODS) Alloys**

- AM can be leveraged to economically produce oxide dispersion strengthened alloys that until now had been cost prohibitive.
- The advanced dispersion coating technique can successfully coat metallic powders with nano-scale ceramics.
- We believe this new manufacturing technique combined with MPEAs opens a new alloy design space for future high temperature alloys

## **LEW-19886-1: “Additively Manufactured Oxide Dispersion Strengthened Medium Entropy Alloys for High Temperature Applications”**

- Thermodynamic models correctly predicted a stable solid solution matrix phase for Alloy X.
- SEM and TEM characterization confirms a uniform dispersion of nano-scale oxides throughout the alloy X – ODS build
- High temperature mechanical testing of Alloy X – ODS reveals surprising and superior results compared to previous ODS alloys produced within this project and conventionally manufactured high temperature alloys.



# Acknowledgments

Questions?



- ASG
- Dave Ellis
- Henry de Groh
- Quynhgiao Nguyen
- Joy Buehler
- Bob Carter
- Pete Bonacuse
- Chris Kantzos
- Cheryl Bowman
- Tim Gabb



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